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THORIUM UTILIZATION PROGRAM

QUARTERLY PROGRESS REPORT FOR THE PERIOD ENDING AUGUST 31, 1974

Prepared under
Contract AT(04-3)-167
Project Agreement No. 53
for the
San Francisco Operations Office
U.S. Atomic Energy Commission

DATE PUBLISHED - OCTOBER 31, 1974

GENERAL ATOMIC

GENERAL ATOMIC COMPANY
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ABSTRACT

This publication is the first of a quarterly series presenting results of work performed under the National HTGR Fuel Recycle Program (also known as the Thorium Utilization Program) at General Atomic Company. Results of work on this program were previously included in a quarterly series on the HTGR Base Program.

The work reported includes the development of unit processes and equipment for reprocessing of High-Temperature Gas-Cooled reactor (HTGR) fuel and the design and development of an integrated line to demonstrate the head end of HTGR reprocessing using unirradiated fuel materials. Work is also described on the evaluation of alternate techniques for fuel reprocessing to surmount possible operating problems with the reference flow sheet and the development of the conceptual design of a target recycle facility to identify the requirements of large-scale recycle of HTGR fuels.

INTRODUCTION

This report covers the work performed by General Atomic Company under U.S. Atomic Energy Commission Contract AT(04-3)-167, Project Agreement No. 53. The work done under this project agreement is part of the program for development of recycle technology for High-Temperature Gas-Cooled Reactor (HTGR) fuels described in the "National HTGR Fuel Recycle Development Program" (ORNL 4702).

The objective of the program is to provide the necessary technology, development, engineering, and demonstration of the steps required in the economic recycle of HTGR fuels utilizing thorium as a fertile material. Work at General Atomic Company is concentrating on the development of reprocessing methods (subtask 110 of the National Program), engineering and economic studies (subtask 310), and the application of recycle technology to the conceptual design of a large-scale recycle facility (subtask 320).

The objectives of subtask 110, Reprocessing Development, are to develop the necessary technology for the construction and operation of a prototype reprocessing facility which will process irradiated fuel materials and to provide the capability for commercial recycle of HTGR fuels. The output of this subtask includes (1) definition of process flow sheets, (2) development of equipment components, and (3) definition of operating data.

The objectives of subtask 310 are to guide the development program from the viewpoint of overall recycle needs and to obtain an economical HTGR fuel recycle method for early recovery and use of bred U-233. Alternate methods and options for reprocessing and refabrication are evaluated, and recommendations are made for possible further experimental development.

The objectives of subtask 320 are to develop a conceptual design of a target-size recycle plant for the reprocessing and refabrication of HTGR fuels and to use the results developed in the preparation of this design to guide the development work. The output of this task is in the form of design criteria, reference process flow sheets, and equipment sizing for a target recycle facility.

CONTENTS

ABSTRACT	iii
INTRODUCTION	v
1. SUMMARY	1
2. FUEL ELEMENT CRUSHING	3
3. SOLIDS HANDLING	10
3.1. Air Classification	10
3.2. Experimental Pneumatic Transport System	10
3.2.1. System Description	10
3.2.2. Status of Work	14
4. FLUIDIZED BED BURNING	15
4.1. Primary Burner	15
4.1.1. 10-cm Primary Burner	15
4.1.2. 20-cm Primary Burner	64
4.1.3. Prototype Primary Burner	70
4.2. Secondary Burner	84
4.2.1. 10-cm Secondary Burner	84
4.2.2. 20-cm Prototype Secondary Burner	103
5. LEACHING	129
5.1. Inspection of Corrosion Coupons Installed in the 13-cm-Diameter Leacher	129
5.1.1. Summary of Results	129
5.1.2. Discussion	130
5.1.3. General Conclusions and Recommendations	145
5.2. Run Summary for Leaching Runs 72-76, 78-81, and 84	145
5.2.1. Summary	145
5.2.2. Run Conditions	145
5.2.3. Results and Discussion	153
5.2.4. Conclusions and Recommendations	156

5.3.	Investigation Into the Potential for U-232 Crossover Into Fissile Particles Under Leach Conditions	156
5.3.1.	Summary	156
5.3.2.	Experimental Procedure	157
5.3.3.	Results and Conclusions	159
6.	SOLVENT EXTRACTION	163
6.1.	Flooding Tests - Solvent Washing	163
6.2.	Uranium-Thorium Coextraction, Thorium Partitioning, Partition Scrubbing, and Uranium Stripping	165
7.	SYSTEMS DESIGN	170
7.1.	Prototype Size Reduction System	170
7.2.	Prototype Primary Burner	171
7.3.	Prototype Secondary Burner	171
7.4.	Prototype Solids Handling	172
7.5.	Prototype Plant Systems Operations	172
7.6.	Remote Operations and Maintenance of Prototype Pilot Line	172
7.7.	E-Building	180
8.	ALTERNATE HEAD-END TECHNIQUES	183
8.1.	Introduction	183
8.2.	Summary of Results	183
8.3.	Conclusions and Recommendations	186
9.	CONCEPTUAL DESIGN OF AN HTGR FUEL COMMERCIAL RECYCLE PLANT . . .	187
	REFERENCES	191

FIGURES

1.	Side view of UNIFRAME crushing system	5
2.	Front view of UNIFRAME crushing system	7
3.	Computer plot of undeformed shape	9
4.	Pneumatic transport system	11
5.	10-cm primary burner	16
6.	Bottom feed system, 10-cm primary burner	17
7.	Top feed system, 10-cm primary burner	18
8.	Size distributions, Run 38, 10-cm primary burner	28
9.	Clamshell cooling system and location of thermocouples, 10-cm primary burner	30

FIGURES (continued)

10.	10-cm primary burner thermowell	31
11.	Cooling air calibration, Run 38, 10-cm primary burner	32
12.	Size distributions, Run 39, 10-cm primary burner	40
13.	Heat transfer calculations, Run 39, 10-cm primary burner	42
14.	Off-gas analysis, Run 42	52
15.	Cooling air calibrations, Run 44, 10-cm primary burner	60
16.	Heat transfer calculations, Run 44, 10-cm primary burner	61
17.	20-cm primary burner	65
18.	20-cm burner with vortex inducer spool	68
19.	Perforated cone and plenum gas distributor	69
20.	Prototype 40-cm burner gas distributor plenum	73
21.	Prototype 40-cm burner remote flange and heater	75
22.	Masrock and Foam-50 insulation	77
23.	Total electrical and thermal loss to coil cooling water as a function of insulation thickness	78
24.	Induction coil parameters as a function of insulation thickness	79
25.	Temperatures for whole bed startup from ambient	80
26.	Temperatures for "rapid dump" startup with tube initially at 900°C	81
27.	20-cm primary burner assembly	83
28.	20-cm primary burner vessel assembly	85
29.	Gas flow, Run 37, 10-cm secondary burner	88
30.	Product size distribution, Run 37, 10-cm secondary burner	91
31.	Product size distribution, Run 38, 10-cm secondary burner	93
32.	Feed size distribution, Run 39, 10-cm secondary burner	94
33.	Product size distribution, Run 39, 10-cm secondary burner	96
34.	Gravity-pneumatic feeder, 10-cm secondary burner	98
35.	Feed size distribution, 20-cm prototype secondary burner	107
36.	Product size distribution, 20-cm prototype secondary burner	108
37.	20-cm secondary burner	117
38.	Tube and filter assembly	119
39.	Distributor plate, 20-cm prototype secondary burner	122
40.	20-cm secondary burner base	124

FIGURES (continued)

41.	Vessel assembly showing semi-remote flanges	127
42.	13-cm-diameter leacher showing position of related equipment and corrosion coupons	134
43.	F-series coupons and mounting bracket	136
44.	Mounting bracket for coupons	137
45.	Positions of coupon brackets as mounted on dip legs of 13-cm-diameter leacher	138
46.	Typical coupons (304L SS) before and after 22 hr of exposure to Thorex - burner ash solution	143
47.	Dissolution of secondary burner ash in presence of whole TRISO fissile particles	146
48.	Particle size distribution in leaching runs	147
49.	Flow sheet for crossover studies	158
50.	Solvent wash column flooding curves for 30% TBP	164
51.	Prototype plant arrangement, burning and crushing (Sheet 1 of 3)	173
51.	Prototype plant arrangement, burning and crushing (Sheet 2 of 3)	175
51.	Prototype plant arrangement, burning and crushing (Sheet 3 of 3)	177
52.	E-building layout	181

TABLES

1.	Summary of 10-cm primary burner runs	19
2.	Operating conditions - Run 37	23
3.	Operating conditions - Run 38	25
4.	Mass balance - Run 28	26
5.	Material properties - Run 38	27
6.	Heat transfer calculations - Run 38	29
7.	Sample analyses - Run 38	34
8.	Heavy metal from broken particles - Run 38	35
9.	Operating conditions - Run 39	36
10.	Mass balance - Run 39	37
11.	Material properties - Run 39	39

TABLES (continued)

12.	Heat transfer calculations - Run 39	41
13.	Sample analyses - Run 39	43
14.	Heavy metals from broken particles - Run 39	44
15.	Comparison of broken particle data	45
16.	Fines data - Runs 40 and 41	47
17.	Temperature profiles - Run 41	49
18.	Fines data - Run 43	50
19.	Comparison of average off-gas CO/CO ₂ ratios	53
20.	Fines elutriation data - Run 42	55
21.	Fines elutriation data - Run 44	57
22.	Heat transfer data - Run 44	58
23.	Heat transfer data - Run 45	62
24.	Comparison of Runs 44 and 45	63
25.	Composition of P-2 type TRISO fertile particle	104
26.	Composition of secondary burner feed	105
27.	Composition of secondary burner product	106
28.	Properties of feed and product	109
29.	U _{mf} and U _t for feed and product	112
30.	Quality of fluidization	113
31.	Slugging fluidization	114
32.	Design conditions	116
33.	Sintered metal filters	118
34.	Selected corrosion data from literature on metal alloys in nitric acid/Thorex solutions	131
35.	Corrosion coupon alloys used in 13-cm-diameter leacher	133
36.	Typical solid size distributions of secondary burner ash	139
37.	Corrosion coupon exposure history in 13-cm-diameter leacher	140
38.	Visual inspection of corrosion coupons after 22 hr of exposure	141
39.	Corrosion rates of coupons installed in 13-cm-leacher after an exposure of 22 hr	144
40.	Leacher operating data, 13-cm-diameter leacher	149
41.	Size distribution of TRISO fissile particles used in leach Runs 72 and 73	150

TABLES (continued)

42.	Chemical analysis of TRISO fissile particles	150
43.	Sample analysis results, 13-cm-diameter leacher	151
44.	Thorium material balance results, 13-cm-diameter leacher . . .	152
45.	Uranium and thorium contents following leaching	160
46.	Flooding data as a function of phase ratio	166
47.	Analytical data and stream flows - Runs 6 and 7	167
48.	Column HETS and flooding data - Runs 6 and 7	168
49.	Total nitrogen analyses by LECO	185

1. SUMMARY

Structural analyses of several components for the full-scale integrated size reduction system have been completed. Modal and dynamic analyses were also performed on the overall system configuration utilizing the GA NASTRAN computer program.

Experimental testing on the air classification of TRISO/TRISO fuel blends has been completed; a GA topical report on these tests will be published in early December 1974. An experimental pneumatic transport system, separate from the equipment associated with the pilot plant facility and prototype line, has been designed and is under evaluation. This test assembly will be utilized for obtaining parametric information on solids transport.

Primary fluidized-bed experimental efforts were concentrated on the 10-cm primary burner. This work consisted of 14 test runs, including two that utilized feed made by crushing half-sized fuel elements loaded with reject FSV fuel rods. Reactivation of the 20-cm primary burner with modifications reported last quarter (Ref. 1) was completed. The design work on the prototype primary burner is progressing; technical design work on the heating, cooling, gas distribution, and off-gas systems is nearly complete.

Secondary fluidized-bed burner experimental efforts were concentrated on shake-down testing of the modified 10-cm secondary burner. The experiments included testing of the induction heating system and alternate operating methods. The conceptual design of the prototype secondary burner is nearly complete and detailed design is started on several of the component parts.

The initial evaluation of corrosion coupons installed in the dissolver has been completed. Experimental testing in the dissolution of crushed, burned-back TRISO fertile particles in the presence of whole SiC coated

fissile particles was continued. During these experimental runs it was established that the presence of whole particles adversely affects the overall batch dissolution rate and the subsequent liquid-solid separation. Evaluation of the problems encountered is continuing.

Preliminary investigations confirmed that the potential for significant crossover of U-232 into the U-235 stream when BISO (or crushed burned-back TRISO) fertile particles are dissolved in the presence of whole TRISO fissile particles is such that the refabrication of recycled U-235 may not be accomplished without remote refabrication. This work was preliminary in nature, and additional testing will be conducted as more definitive results are required.

Solvent extraction experimental testing consisted of two run sets utilizing a single-column solvent extraction system and five run sets utilizing the newly installed two-column system. Initial data on flooding tests, solvent extraction washing, extraction, partition, partition scrub, and uranium stripping runs were received.

Evaluation of remote maintenance features within the prototype size reduction system was expanded in scope and is currently in progress. The conceptual and final designs of the prototype primary and secondary burners have been completed, and the detailed design drawings are in preparation. The demonstration of prototype component configuration and adaptability for remote installation, operation, maintenance, and removal and/or disassembly will be performed utilizing the prototype pilot line to simulate hot cell operating conditions.

Efforts were made to analyze the nitrogen content of fresh fuel samples in order to correlate nitrogen contamination with levels of carbon-14 released from irradiated HTGR fuel. Analytical results obtained were generally inconclusive and a direct approach to measurement of actual carbon-14 levels appears to be indicated.

A study of the conceptual design of a commercial-scale recycle facility for HTGR fuel was initiated. Design has progressed through design criteria definition and system descriptions.

2. FUEL ELEMENT CRUSHING

Three tons of H-327 graphite have been crushed to $<3/16$ in. (ring size) for use as feed material for fluid-bed burner experiments.

Structural analyses of several components for the full-scale integrated size reduction system, designated UNIFRAME, have been completed. The components of this system are identified in Figs. 1 and 2. Modal and dynamic analyses were also performed on this configuration using the NASTRAN computer program (Ref. 2). A computer plot of the "undeformed shape" is shown in Fig. 3. Figure 3 differs somewhat from Figs. 1 and 2 in that the frame configuration in Figs. 1 and 2 does not contain cross bracing. The ultimate decision regarding the use of cross bracing depends to a large extent on the degree to which the present system is to demonstrate remote maintenance features.

Fig. 1. Side view of UNIFRAME crushing system

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PRELIMINARY DESIGN

ITEM	PART NO	DESCRIPTION	QTY	UNIT
1	212100	HINGE SECOND STAY JAW	1	ASSY
2	212132	UPPER ANCHOR SEC STAY JAW	1	ASSY
3	212130	LOWER ANCHOR SEC STAY JAW	1	ASSY
4	212120	SECONDARY PUSHING BLOCK SUPPORT	1	ASSY
5	212112	SECONDARY PILLOW BLOCK RIGHT	1	ASSY
6	212110	SECONDARY PILLOW BLOCK LEFT	1	ASSY
7	212080	HINGE, ARM STAY JAW	1	ASSY
8	212038	DOUBLE ARM HINGE STAY JAW	1	ASSY
9	212034	UPPER ANCHOR ARM STAY JAW	1	ASSY
10	212032	MIDDLE ANCHOR ARM STAY JAW	1	ASSY
11	212030	LOWER ANCHOR ARM STAY JAW	1	ASSY
12	212070	PRIMARY PUSHING BLOCK SUPPORT	1	ASSY
13	212070	PRIMARY PILLOW BLOCK	1	ASSY
14	212000	UNIFRAME	1	WEB-35 2120 2120 2120
15	211100	UNIFRAME ASSEMBLY	1	WEB-35 2120 2120 2120

Fig. 2. Front view of UNIFRAME crushing system

Fig. 3. Computer plot of undeformed shape

3. SOLIDS HANDLING

3.1. AIR CLASSIFICATION

Experiments on classification of TRISO-TRISO fuel blends have been completed. It is expected that the GA topical report on these experiments will be published during the next quarter (Ref. 3). This report will document the efficiency of separation by pneumatic classification and establish the relationship of the efficiency to: (1) air flow rate, (2) solids feed rate, (3) height of the separation column, (4) position of the feed zone, and (5) shape of the feed size distribution. The crossover between the fissile and the fertile streams will also be related to this efficiency. For comparison, the efficiency of separation for sieving will be provided, including the effect of screen blinding.

3.2. EXPERIMENTAL PNEUMATIC TRANSPORT SYSTEM

3.2.1. System Description

An experimental pneumatic transport system, separate from the equipment associated with the pilot plant facility, will be assembled to obtain information on equipment operation and the effects of process variables. The system (shown schematically in Fig. 4) will be capable of testing a variety of equipment and modes of operation with many types of materials including (1) crushed graphite, (2) SiC coated fuel particles plus graphite, and (3) broken SiC shells plus ThO_2 powder. The experimental system consists of six basic subsystems: (1) material feed vessel, (2) material feeders, (3) transport piping, (4) fines filter vessel, (5) material receiver, and (6) positive displacement pump (motive force).

Fig. 4. Pneumatic transport system

The material feed vessel is sized to contain a sufficient quantity of each of the types of material for periods of time that are compatible with run duration at plant design rates. Material level indicators will be installed and tested. A filtered pressure tap will provide information on transport conditions and air leakages within the vessel during operation. The vessel will be flanged for changing the bottom section to fit the various feeders and for altering the cone angles. A solids fill port will be provided for weighed batch charging, as well as a material recycle system for continuous running.

The material feeders will be selected on their relative ability to meet the following criteria: positive feed and shutoff, metering ability and accuracy, completeness of discharge, remote operability, maintenance of particle integrity, non-heat sensitivity, and ability to avoid choking or flooding. Several commercially available feeder devices will be tested, and others will be specifically designed for testing. The devices under consideration are screw conveyors, rotary feeders, vibratory feeders, venturis, angle of repose feeders, flow nozzles, air slides, and slot flow feeders. Where practicable, differential pressure data will be obtained across the material pickup points of each device tested.

Interchangeable transport piping of 1/2, 1, and 2 in. o.d. tubing, with several pipe diameter to sweep radius ratios, will be provided for studying the effects on material holdup, solids-to-air loading ratios, flow rates, saltation velocity, choking velocity, pressure drop, and particle breakage. The piping will be fitted with several types of quick disconnect sanitary-type connectors to test them for material holdup, leakage, and ease of operation. Strategically located filtered pressure taps and sight tubes will provide pressure data and visual observation of material movement. Testing the effects of varying horizontal and vertical arrangements and distances between bends, as well as the effect of reducing linear velocity at entry into the receiver via a smooth transition to a tube of larger cross-sectional area, will be accomplished by alterations to the basic piping layout.

The fines filter vessel is designed to separate entrained fine powders from the off-gas stream with maximum practicable efficiency while maintaining a low pressure drop across the filter system. The vessel will contain nine filter elements. Each element will contain a cluster of six 3/4-in.-diameter, 36-in.-long sintered metal tubes of 5- μ m porosity, which provide a relatively large filter surface area in the occupied space. Accumulated fines on the filters will be periodically removed by a pulse of high-pressure reverse air flow (blow-back) through individual jets located above each filter element. The duration and frequency of the pulses will be controlled by an adjustable multicircuit cam timer, which actuates solenoid valves allowing high-pressure air from a surge tank to dislodge the accumulated fines. The filter vessel will be constructed of clear acrylic for visual observation of the performance of the filters and will have strategically located filtered pressure taps for measurement of the pressure drop across the filters. An adjustable timer will be used to determine the optimum pulse duration and frequencies for each type of material handled.

The material receiver will have several tangential entry points for ease in connecting the various transport line sizes being tested. It will be fitted to a weigh system for material accountability and tests on the capabilities of the weigh system. For continuous runs a material recycle system consisting of sequentially operated isolation valves (punch type) is provided.

The motive force for transport will be provided by a positive displacement air pump, which will be capable of various negative pressure ranges and flow capacities. Further alterations of the flow will be accomplished by a manual air bleed just before the suction side of the pump. Potential overloading of the pump will be avoided via an automatic bypass valve. Measurements of the material passing through the sintered metal filters, as well as protection of the pump, will be accomplished by an in-line absolute filter. Pressure taps located before and after the pump will yield data on pressure drops at the various run conditions.

3.2.2. Status of Work

An outline of the functions of the system and its subsystems, the requirements of the system, and potential problem areas has been prepared and reviewed. Design of the system, including detailed design of some components and equipment specifications, is approximately 85% complete. Procurement requisitions have been written for approximately 75% of the necessary items. Feeder and pump vendors have been contacted and are submitting information on capabilities and costs of these items. Other vendors (filter manufacturers, pneumatic transport system suppliers, etc.) have been contacted for information on limitations and applications of specific items. Work is continuing toward preparation of a procurement package.

4. FLUIDIZED BED BURNING

4.1. PRIMARY BURNER

4.1.1. 10-cm Primary Burner

4.1.1.1. Summary

Fourteen runs were made utilizing the 10-cm primary burner during the last quarter. Emphasis was placed on component performance and the startup technique. The present system is shown in Figs. 5 through 7. The burner vessel is fabricated from Hastelloy-X. The runs are summarized in Table 1.

Evaluations of several areas were completed. Although augers perform satisfactorily for unmixed feed systems, it was determined that feeding mixed fresh feed (crushed graphite and fuel particles) and recycled fines is not a satisfactory feed arrangement when using augers. The addition of secondary oxygen was found to have no significant effect on burner operation, although the axial temperature profile was more isothermal. It was confirmed that the use of CO_2 as a diluent has no significant impact on operation.

4.1.1.2. Run 34A

The primary objective of Run 34A was to determine the ability to build up the initial bed using bottom fresh feed only and not top feed as was done in most previous work. Problems arose when the bottom addition of fresh feed (<3/16 in.) tended to extinguish the bed because of the relatively high heat capacity of the cold feed and the small amount of

Fig. 5. 10-cm primary burner

Fig. 6. Bottom feed system, 10-cm primary burner

Fig. 7. Top feed system, 10-cm primary burner

TABLE 1
SUMMARY OF 10-CM PRIMARY BURNER RUNS

Run	Objective	Fines Recycle	Secondary Oxygen	Results
34A	Build up initial bed using bottom fresh fuel only	Yes	No	Cone break-through from agglomeration resulting from operation of torch with fuel particles in bed.
34B	Build up initial bed using top feed graphite	Yes	No	Successful startup - run terminated due to poor material flow in recycle hopper.
35	Test recycle auger aeration system and determine feasibility of long-term operation with fines recycle	Yes	No	Long-term operation using fines recycle in present system determined to be very difficult due to feed system design.
36	Simulate operating conditions for future FSV reject rod runs	No	No	Run terminated due to severe bed segregation after fresh feed was introduced too soon.
37	Simulate operating conditions for future FSV reject rod runs	No	No	Successful run after delaying start of fresh feed flow until after complete initial bed addition.
38	FSV reject rod run 1 - determine fuel particle breakage directly related to burner operation	No	No	Particle breakage due to both crushing and burning was approximately 3 to 4% of fissile particles and 17% of fertile particles.
39	FSV reject rod run 2 - determine fuel particle breakage directly related to burner operation	No	No	Particle breakage due to both crushing and burning was approximately 2.5% of fissile particles and 7% of fertile particles. Differences from previous run attributed to smaller diameter and stronger SiC layers for fuel particles in this run.
40-41	Investigate effects of secondary oxygen inlet on burner operation	Yes	Yes	Lengthening of burning zone and improved utilization of heat transfer surface. No significant effects on fines elutriation due to secondary oxygen. Overall heat transfer coefficients determined for laminar flow of cooling air.

TABLE 1 (Continued)

Run	Objective	Fines Recycle	Secondary Oxygen	Results
42	Determine effects on burner operation of using CO ₂ in place of N ₂ as fluidization diluent	Yes	No	Use of CO ₂ did not noticeably affect corrected ^(a) off-gas CO/CO ₂ ratio. No change in fines elutriation characteristics due to use of CO ₂ diluent. The only appreciable effects of CO ₂ diluent on operation were slightly faster temperature transients and a larger startup period.
43A	Investigate effects of secondary oxygen inlet on burner operation	Yes	Yes	Run terminated early when flow of feed in recycle augers backed up and resulted in binding.
43B	Investigate effects of secondary oxygen inlet on burner operation	Yes	Yes	Secondary oxygen usage relative to runs 40 and 41 was increased and resulted in no change in fines elutriation characteristics. Fines elutriation is largely dependent on fluidizing gas velocity and weight of fines in feed.
44	Determine overall heat transfer coefficients and maximum possible burn rate using increased (turbulent) cooling air flow	Yes	No	Maximum attainable burn rate was 57 g C/min. Overall heat transfer coefficients were obtained and calculations showed that the controlling heat transfer resistance was that of the outer burner film. As cooling air increases, this resistance becomes more controlling.
45	Determine overall heat transfer coefficients and maximum possible burn rate using increased (turbulent) cooling air flow	Yes	Yes	Maximum attainable burn rate was 57 g C/min. Use of secondary oxygen lengthened the burning zone such that the distribution of heat removal was shifted upward in the burner.

(a) Corrected to exclude CO₂ entering burner in fluidizing gas.

reaction heat generated during initial bed ignition. After repeated unsuccessful attempts to reignite the bed with the CO/O₂ torch, the bed was emptied and the startup process was repeated. Apparently, fuel particle removal was incomplete because a cone breakthrough occurred after the second startup. Further analysis showed fuel particle agglomeration, which apparently resulted from use of the CO/O₂ torch with fuel particles in the burner.

4.1.1.3. Run 34B

Run 34B was made to investigate the startup technique using an initial top feed of graphite. A mixture of 3.5 kg graphite (-710, +355 μ m) and coke (6:1 ratio) was top fed immediately after bed ignition, and bottom fresh feed (<3/16 in.) was begun approximately 5 min later. The initial bed was built up smoothly without incident by this technique. The run was eventually terminated when poor flow of mixed fresh feed and recycled fines developed in the recycle hopper such that the recycle augers could not transport feed fast enough. Dismantling of the system showed that "rat-holing" was occurring in the hopper. It was decided to install aeration screens along the sides of the recycle hopper to provide sufficient N₂ flow to prevent occurrence of this "rat-holing." Two extremely fine (Dynapore) screens were welded onto opposite sides of the hopper such that the N₂ flow through and into the hopper would provide a fluidizing medium for the feed around the periphery.

4.1.1.4. Run 35

Following several successful tests of the aeration system, it was used during Run 35. The objective of Run 35 was to determine if long-term operation of the recycle auger feed system (Refs. 4,5) was feasible since the FSV reject rod program experiments were of immediate concern and required long-term operation. Run 35 lasted 59 min and underwent a controlled shutdown when the feed supply was exhausted. The average burn

rate was 32.8 g C/min. Throughout the entire run, the recycle auger speed had to be periodically increased in order to transport sufficient feed. This was to be expected since the bulk density of the recycle feed decreased as more fines entered the hopper. During the last several hours, it was necessary to operate the motor at the maximum of 29 rpm. When the system was dismantled after the run, the recycle hopper was found to be completely full and backed up into the lines feeding the hopper. It was apparent that with the given system and mode of operation, long-term operation with recycle of fines would have been extremely difficult, if not impossible.

4.1.1.5. Run 36

Run 36 was performed in the slightly modified burner (Ref. 6) using bottom fresh feed and no fines recycle. The objective of this run was to simulate operating conditions to be used in the subsequent FSV reject rod experiments and to demonstrate long-term reliability of the operation. Run 36 was performed by top feeding an initial bed of 4.5 kg of graphite ($-710, +355 \mu\text{m}$) and coke (8:1 ratio). Approximately 15 min into the run, bottom fresh feed ($<3/16$ in.) was begun. After 105 min of operation and a bed buildup to 3.5 kg, evidence of bed segregation appeared as the core exhibited a "zebra" hot and cold effect. The run was immediately terminated. Subsequent analysis of the final bed showed it to consist mainly of very large pieces of graphite and fuel particles. This result indicated that building up the initial bed with relatively large material ($<3/16$ in.) was an unsafe practice in that it led to preferential burning and an eventual undesired bed size distribution.

4.1.1.6. Run 37

Run 37 was also performed in the slightly modified burner with bottom feed and no fines recycle. The objective for this run was the same as for Run 36 except that the bottom fresh feed was not begun until well into the

run when the top feed initial bed addition had been completed. After 430 min of smooth and successful operation at the average conditions listed in Table 2, the run was terminated when the bed was allowed to burn itself out.

TABLE 2
OPERATING CONDITIONS - RUN 37

Average feed rate (all sources), g/min	69.8
Average burn rate, g C/min	32.8
Average fines carryover rate, g C/min	
Cyclone	23.6
Filter.	0.4
Average product take-off, g/min.	7.6
Final bed (kg)	2.35

4.1.1.7. Run 38

Run 38 was the first of two runs made in the 10-cm primary burner utilizing crushed material from FSV-type (half-size) fuel blocks loaded with reject fuel rods as feed. The fuel particles used in this run represented one of the extreme size combinations of FSV-type fuel in that the fuel was selected for minimum overlap in particle size between the fissile and fertile types. The main objectives were to determine fuel particle breakage directly related to burner operation and to reduce the crushed fuel block to a material suitable for air classification of the fissile and fertile fuel particles. The burner was operated with bottom fresh feed (<3/16 in.) and no recycle of elutriated fines. Ignition occurred at 14:45 (6/4/74) and shutdown was at 05:12 (6/5/74) after 867 min of operation. Eight kilograms of graphite (~1000, +355 μ m) and coke (4.3:1 ratio) were top fed initially before beginning bottom fresh feed. After approximately 3 hr, the bed was built up to the desired amount of 4 to 4.5

kg. The rotary product take-off valve (Ref. 7) was switched to automatic 163 min into the run and maintained the desired bed weight thereafter. The total cone gas flow ($O_2 + N_2$) was maintained at 85 to 90 SLPM (standard liters per minute) during most of the run.

Table 3 summarizes the operating conditions used during the run, and Table 4 gives the overall mass balance. The average burn rate was 37.2 g C/min, and the average fines elutriation rate was 22.2 g C/min. This fines elutriation rate was calculated over the entire run. Since the first 80 min of operation was performed with top feed much smaller than the fresh feed, the fines elutriation rate over this period was probably higher than 22.2 g C/min. A more realistic value over the last 790 min of the run would be 18 to 20 g C/min. The average product take-off (after 163 min) was 13.8 g/min, and the average fresh feed rate (after 80 min) was 70.5 g/min.

Table 5 summarizes the measured material properties and Fig. 8 shows the size distributions of the fresh feed, fines, and product. The size distribution plot for the fines and visual inspection show that only a small part of the sample was fuel particles. Based on this analysis (including graphite-particle separation from screen samples), it was estimated that less than 1 g of fuel particles was carried over, along with more than 19 kg of fines, during the run.

Overall heat transfer coefficients calculated at different times during the run are shown in Table 6. The clamshell cooling system is shown schematically in Fig. 9. The system consists of one 18-in.-long and three 12-in.-long clamshells. The annular space through which the cooling air flows is 3/4 in. wide and surrounds all but 6 in. of the 60-in.-long, 4.5-in.-o.d. burner vessel. The thermocouples from which the operating temperatures were determined are shown in Figs. 9 and 10. Figure 11 shows the cooling air calibrations for each of the clamshells for conditions existing in the laminar flow region ($Re < 2000$).

TABLE 3
OPERATING CONDITIONS - RUN 38

Time (min)	Maximum Bed Temp (°C)	Total Gas Flow (SLPM)	O ₂ Flow (SLPM)	Bed Size (kg)	Superficial Velocity (cm/sec)		Burn Rate (g C/min)
					At Thermo- couple 1	At Thermo- couple 8	
60	890	81	69	2.1	71.2	56.7	38.3
90	894	79	64	2.4	69.4	54.9	36.1
150	895	84	64	3.4	73.0	53.4	36.9
215	887	86	66	4.4	73.4	55.7	38.7
285	911	86	60	4.2	75.0	48.3	34.5
365	915	86	61	4.3	75.2	49.9	34.8
490	918	86	60	4.2	75.4	44.7	34.5
585	923	86	60	4.2	75.8	44.3	34.4
670	926	89	60	4.1	78.7	46.7	34.8
755	910	89	61	4.1	77.6	45.3	34.4
820	920	88	61	4.1	77.4	45.4	35.4
860	948	110	50	2.3	101.3	54.4	22.9
867	800	60	0	2.0	48.7	28.3	0

TABLE 4
MASS BALANCE - RUN 38

Startup coke	500	Total product	10,089
Top bed addition graphite		Cyclone fines	18,506
-710, +355 μm	2,000	Filter fines	718
-1000, +500 μm	3,700	Final bed	2,031
-1000, +355 μm	2,000	Unused feed	1,281
Coke	1,500		
Fresh feed	<u>55,190</u>		<u> </u>
TOTAL IN		TOTAL OUT	
	64,890 g		32,625 g

32,265 g carbon burned by material balance

$$\text{Burn Rate} = \frac{32,265 \text{ g C}}{867 \text{ min}} = 37.2 \frac{\text{g C}}{\text{min}}$$

TABLE 5
MATERIAL PROPERTIES - RUN 38

Material	Density (g/cm ³)	Tap Density (g/cm ³)	Angle of Repose ^(a) (deg)	Average Particle Size ^(b) (μm)
Fresh feed	1.09	1.43	39	1300
Product	1.51	1.67	29	475
Cyclone fines	0.50	0.78	33	86

^(a) Maximum angle (with vertical) at which material will flow freely.

^(b) 50 wt % greater than this size.

Fig. 8. Size distributions, Run 38, 10-cm primary burner

TABLE 6
HEAT TRANSFER CALCULATIONS - RUN 38

Cooling Air Setting (%)	Clamshell	Clamshell Air Flow (at 25°C) (ft ³ /min)	Clamshell Heat Removal, Q		ΔT_{ln} (°F)	$\bar{U} \left(\bar{U} = \frac{Q}{A \Delta T_{ln}} \right)$ (Btu/hr-ft ² -°F)	Total Heat By Combustion (Δ hr x Btu/hr)
			Btu/hr	%			
20	1 (bottom)	7.5	6,736	46.4	1,136	5.0	55,000
	2	8.0	5,056	34.8	1,144	3.8	
	3	6.3	1,650	11.4	925	1.5	
	4 (top)	8.0	1,081	7.4	627	1.0	
30	1	9.5	8,038	46.0	1,176	5.8	57,000
	2	9.5	5,728	32.8	1,159	4.2	
	3	9.5	2,370	13.5	926	2.2	
	4	9.7	1,351	7.7	626	1.2	
40	1	11.9	9,179	46.2	1,205	6.5	57,000
	2	10.8	5,905	29.7	1,168	4.3	
	3	14.8	3,200	16.1	924	2.9	
	4	12.7	1,584	8.0	624	1.4	
50	1	14.8	11,044	44.7	1,199	7.8	56,000
	2	12.6	7,047	28.5	1,198	5.0	
	3	18.7	4,315	17.5	985	3.7	
	4	16.4	2,284	9.3	655	2.0	
60	1	17.3	12,624	46.9	1,223	8.8	56,000
	2	14.5	7,567	28.1	1,189	5.4	
	3	20.6	4,283	15.9	934	3.9	
	4	20.6	2,441	9.1	629	2.2	
100	1	19.7	13,516	48.5	1,252	9.2	47,000
	2	17.3	7,517	27.0	1,077	5.9	
	3	22.3	3,848	13.8	859	3.8	
	4	27.7	2,995	10.7	609	2.8

Fig. 9. Clamshell cooling system and location of thermocouples, 10-cm primary burner

Fig. 10. 10-cm primary burner thermowell

Fig. 11. Cooling air calibration, Run 38, 10-cm primary burner

Tables 7 and 8 show the results of broken fuel particle analyses performed on samples taken at various stages of the burning operation. Samples from the burner feed, product, fines, and final bed were analyzed for burnable carbon, heavy metal content, and broken fuel particles. As shown in Table 8, most of the heavy metal released from broken particles was elutriated and ultimately ended up in the fines. Since only a small number of fuel particles was detected in the cyclone fines during a size distribution analysis, it was evident that practically all of the elutriated heavy metal had been oxidized to finer material in the burner and subsequently elutriated. Total particle breakage due to both the crushing and burning operations was approximately 3 to 4% of the fissile particles and 17% of the fertile particles.

4.1.1.8. Run 39

Run 39 was the second of two runs utilizing FSV-type (half-size) fuel blocks loaded with reject fuel rods as feed. The fuel particles used in this run represented the opposite extreme of Run 38 size combination in FSV-type fuel, i.e., maximum overlap in the diameters of the fissile and fertile particle types. The operating conditions and experimental procedure were similar to those of Run 38. Ignition occurred at 13:16 (6/19/74) and shutdown was at 03:22 (6/20/74) after 846 min of operation. During this run, 7.5 kg of graphite (-1400, +355 μm , 15% >1000 μm) was top fed initially before beginning bottom fresh feed; the initial bed was built up to the desired amount of 4 to 4.5 kg in 150 min. The rotary product take-off valve was switched to automatic 152 min into the run and maintained the desired bed weight thereafter. The total cone gas flow ($\text{O}_2 + \text{N}_2$) was maintained at 85 to 90 SLPM during most of the run.

Table 9 summarizes the operating conditions for the run, and Table 10 gives the overall mass balance. The average burn rate was 36.6 g C/min, and the average fines elutriation rate was 19.8 g C/min. As in Run 38, the

TABLE 7
SAMPLE ANALYSES - RUN 38

Burner Material	Time (min)	Weight (g)	Average Rate (g/min)	Sample Weight (g)	Burnable Carbon (wt %)	Sample U ^(a) (wt %)	Sample Th ^(a) (wt %)	Sample Broken Particles ^(b)	
								No.	% of Sample
Product									
1	163-240	248	3.2	--	--	--	--	--	--
2	240-309	884	12.8	--	--	--	--	--	--
3	309-356	1,146	24.4	--	--	--	--	--	--
4	356-405	1,165	23.8	--	--	--	--	--	--
5	405-490	1,259	14.8	20.308	24.37	0.015	1.497	186	0.151
6	490-559	1,015	14.7	--	--	--	--	--	--
7	559-615	729	13.0	20.289	20.51	0.026	2.440	189	0.147
8	615-675	879	14.7	--	--	--	--	--	--
9	675-735	878	14.6	20.229	18.64	0.020	2.148	174	0.132
10	735-795	881	14.7	20.390	22.36	0.015	1.630	180	0.143
11	795-845	611	12.2	20.149	21.63	0.020	1.975	150	0.119
Final bed	--	2,031	--	20.108	9.10	0.004	1.763	192	0.132
Fresh feed	--	55,190	70.5	126.04	81.25	--	--	320	0.170
Filter fines									
1	--	--	--	52.651	98.61	0.013	0.791	--	--
2	--	--	--	53.734	98.52	0.013	0.775	--	--
Cyclone fines									
1	--	--	--	50.103	97.09	0.038	1.939	--	--
2	--	--	--	50.154	97.49	0.031	1.702	--	--

(a) Determined by leaching at analytical laboratory; U and Th content in fines resulted primarily from broken particles in the bed since the number of particles in the fines was extremely small.

(b) Determined by QC laboratory. Approximately 8000 particles/g of particles.

TABLE 8
HEAVY METAL FROM BROKEN PARTICLES^(a) - RUN 38

Source	Total U (g) (b)	Total Th (g) (b)	Broken Particles(c)					
			Fissile			Fertile		
			Number	Number per kg Fissile Particles in Feed	% of Fissile Particles in Feed ^(d)	Number	Number per kg Fertile Particles in Feed	% of Fertile Particles in Feed ^(d)
Cyclone fines	6.383	337.20	1,850,000	392,500	2.90	972,000	180,900	10.07
Filter fines	0.093	5.63	27,100	5,800	0.04	16,400	3,000	0.17
Products ^(e)	2.0	190.0	580,000	123,000	0.90	569,000	106,000	5.90
Final bed	<u>0.073</u>	<u>35.81</u>	<u>21,200</u>	<u>4,500</u>	<u>0.03</u>	<u>111,000</u>	<u>20,700</u>	<u>1.15</u>
Total	8.549	568.64	2,478,300	525,800	3.87	1,668,400	310,600	17.29

(a) Values are averaged from small representative samples.

(b) Determined by leaching at analytical laboratory.

(c) Broken particles from which heavy metals came - based on analytical leaching analysis.

(d) Revised to give percent of particles actually entering burner in feed and not percent of total particles in block.

(e) Estimated since not all product samples were analyzed.

TABLE 9
OPERATING CONDITIONS - RUN 39

Time (min)	Maximum Bed Temp (°C)	Total Gas Flow (SLPM)	O ₂ Flow (SLPM)	Bed Size (kg)	Superficial Velocity (cm/sec)		Burn Rate (g C/min)
					At Thermo- couple 1	At Thermo- couple 8	
50	905	79	63	2.0	70.4	39.6	36.2
100	905	81	65	2.5	71.8	47.9	37.6
150	910	82	65	3.8	71.9	51.3	38.0
230	916	84	63	4.0	73.8	46.8	37.6
350	923	84	60	4.1	74.4	45.4	35.5
420	934	87	61	4.0	77.6	46.0	36.0
510	923	84	58	4.0	74.3	44.5	34.3
600	924	84	58	4.0	74.3	43.4	34.1
690	928	86	57	4.0	76.4	44.0	33.3
780	924	86	57	4.0	76.1	44.5	33.3
830	933	92	50	2.6	83.4	47.8	29.5
842	898	83	60	2.3	73.3	41.9	34.4
846	830	50	0	2.2	41.6	23.9	0

TABLE 10
MASS BALANCE - RUN 39

Startup coke	500	Total products	9,853
Top bed addition graphite		Cyclone fines	16,103
-1000, +355 μm	6,375	Filter fines	666
-1400, +1000 μm	1,125	Final bed	2,200
Fresh feed	<u>52,990</u>	Unused feed	<u>1,211</u>
TOTAL IN 60,990 g		TOTAL OUT 30,033 g	

30,957 carbon burned by material balance

$$\text{Burn Rate} = \frac{30,957 \text{ g C}}{846 \text{ min}} = 36.6 \frac{\text{g C}}{\text{min}}$$

fines elutriation was higher during the 100 min of initial bed addition, and thus a more realistic value for the last 750 min would be approximately 18 g C/min. The average product take-off while the take-off device was in operation was 14.1 g/min, and the average fresh fuel rate (after 70 min) was 70.0 g/min.

Table 11 summarizes the measured material properties, and Fig. 12 shows the size distributions of the fresh feed, fines, and final product. Overall heat transfer coefficients ($\bar{U}$) were calculated at different times during the run and are shown in Table 12. In this run, as in previous runs, entirely adequate cooling was achieved with a laminar flow of cooling air. Comparison of data for Runs 38 and 39 shows the overall heat transfer coefficients to be generally higher in Run 39 due to the higher instantaneous burn rates. Figure 13 shows the effect of air flow on $\bar{U}$ in Run 39.

Tables 13 and 14 show the results of broken fuel particle analyses performed on samples taken at various stages of the burning process. Samples from the burner feed, product, fines, and final bed were analyzed for burnable carbon, heavy metal content, and broken particles. As in Run 38, a size distribution analysis of the fines showed that only a small number of fuel particles was actually elutriated and that most of the heavy metal in the fines resulted from oxidation in the bed to finer material and subsequent elutriation. Total particle breakage due to both the crushing and burning operations was approximately 2.5% of the fissile particles and 7% of the fertile particles entering the burner.

An overall summary of particle breakage results from both the crushing and burner operations of the two reject rod runs is presented in Table 15. The percent of particle breakage attributable to burner operation alone increased for the fissile particles and decreased for the fertile particles from the first run to the second. The difference in breakage is attributed

TABLE 11
MATERIAL PROPERTIES - RUN 39

Material	Density (g/cm ³)	Tap Density (g/cm ³)	Angle of Repose ^(a) (deg)	Average Particle Size ^(b) (μm)
Fresh feed	0.95	1.12	38	1175
Product	1.78	1.86	29	542
Cyclone fines	0.49	0.69	41	86

(a) Maximum angle (with vertical) at which material will flow freely.

(b) 50 wt % greater than this size.

Fig. 12. Size distributions, Run 39, 10-cm primary burner

TABLE 12
HEAT TRANSFER CALCULATIONS - RUN 39

Cooling Air Setting (%)	Clamshell	Clamshell Air Flow (at 25°C) (ft ³ /min)	Clamshell Heat Removal, Q		ΔT_{in} (°F)	$\bar{U} \left(\bar{U} = \frac{Q}{A \Delta T_{in}} \right)$ (Btu/hr-ft ² -°F)	Total Heat By Combustion (Δ hr x Btu/hr)
			Btu/hr	%			
0	1 (bottom)	5.6	4,599	36.2	1,145	3.4	60,500
	2	7.3	4,781	37.6	1,179	3.4	
	3	5.2	2,303	18.1	1,047	1.9	
	4 (top)	7.0	1,033	8.1	797	0.7	
10	1	5.7	4,894	36.6	1,160	3.6	59,000
	2	7.4	4,938	37.0	1,192	3.5	
	3	5.2	2,076	15.5	1,009	1.7	
	4	7.1	1,461	10.9	734	1.1	
20	1	7.5	6,471	44.6	1,155	4.8	58,500
	2	8.0	5,222	35.9	1,131	3.9	
	3	6.3	1,650	11.4	925	1.5	
	4	8.0	1,181	8.1	627	1.1	
30	1	9.5	8,157	42.4	1,153	6.0	57,500
	2	9.5	6,557	34.0	1,147	4.9	
	3	9.5	2,923	15.2	954	2.6	
	4	9.7	1,613	8.4	645	1.4	
40	1	11.9	10,044	42.6	1,157	7.4	57,500
	2	10.8	6,871	29.1	1,170	5.0	
	3	14.8	4,585	19.4	956	4.1	
	4	12.7	2,086	8.9	643	1.8	
50	1	14.8	12,585	44.7	1,176	9.1	57,500
	2	12.6	7,806	27.8	1,171	5.7	
	3	18.7	5,210	18.5	950	4.7	
	4	16.4	2,523	9.0	637	2.2	
60	1	17.3	14,309	45.6	1,180	10.3	58,000
	2	14.5	8,682	27.7	1,164	6.3	
	3	20.6	5,396	17.2	932	4.9	
	4	20.6	2,998	9.5	632	2.7	
70	1	18.4	15,110	45.1	1,182	10.9	58,000
	2	16.2	9,531	28.4	1,169	6.9	
	3	21.5	5,364	16.0	923	4.9	
	4	24.8	3,506	10.5	626	3.2	
100	1	19.7	16,219	45.5	1,187	11.6	58,500
	2	17.3	10,179	28.5	1,241	7.0	
	3	22.3	5,424	15.2	929	5.0	
	4	27.7	3,858	10.8	623	3.5

Fig. 13. Heat transfer calculations, Run 39, 10-cm primary burner

TABLE 13
SAMPLE ANALYSES - RUN 39

Burner Material	Time (min)	Weight (g)	Average Rate (g/min)	Sample Weight (g)	Burnable Carbon (wt %)	Sample U ^(a) (wt %)	Sample Th ^(a) (wt %)	Sample Broken Particles ^(b)	
								No.	% of Sample
Product									
1	152-269	1,031	8.8	20.185	74.44	0.017	0.233	26	0.126
2	269-374	1,447	13.8	20.225	13.42	0.072	2.866	91	0.130
3	374-449	1,491	19.9	20.172	35.95	0.007	0.378	69	0.134
4	449-524	1,153	15.4	20.286	35.67	0.023	0.946	75	0.144
5	524-584	881	14.7	20.210	39.58	0.012	0.574	58	0.119
6	584-664	1,168	14.6	23.248	43.13	0.013	0.591	44	0.083
7	664-749	1,445	17.0	20.185	37.68	0.010	0.517	109	0.217
8	749-826	858	11.4	20.297	34.67	0.009	0.562	46	0.087
9 (tube)	826-846	378	--	19.586	23.87	0.019	0.959	52	0.087
Final bed									
1	--	2,200	--	20.173	7.95	0.006	0.635	89	0.120
2	--	2,200	--	20.215	6.16	0.005	0.582	149	0.196
Fresh feed	--	52,990	69.0	85.463	83.74	--	--	135	0.242
Filter fines									
1	--	--	--	50.100	99.14	0.0127	0.465	--	--
2	--	--	--	50.473	99.10	0.0119	0.493	--	--
3	--	--	--	49.274	99.14	0.0113	0.478	--	--
Cyclone fines									
1	--	--	--	51.213	98.46	0.0312	0.995	--	--
2	--	--	--	50.298	98.42	0.0329	1.028	--	--
3	--	--	--	51.021	98.82	0.0243	0.738	--	--

(a) Determined by leaching at analytical laboratory.

(b) Determined by QC laboratory. Approximately 4000 particles/g of particles.

TABLE 14
HEAVY METALS FROM BROKEN PARTICLES (a) - RUN 39

Source	Total U (g) (b)	Total Th (g) (b)	Broken Particles (c)					
			Fissile			Fertile		
			Number	Number per kg Fissile Particles in Feed	% of Total into Burner	Number	Number per kg Fertile Particles in Feed	% of Total into Burner
Cyclone fines	5.161	162.88	448,000	106,000	1.69	588,000	97,500	4.24
Filter fines	0.080	3.20	7,000	1,600	0.03	12,000	2,000	0.09
Products	2.183	88.30	186,000	44,000	0.70	330,000	54,700	2.38
Final bed	<u>0.121</u>	<u>13.39</u>	<u>10,500</u>	<u>2,500</u>	<u>0.04</u>	<u>54,000</u>	<u>8,900</u>	<u>0.39</u>
Total	7.500	267.77	651,000	154,100	2.46	984,000	163,100	7.10

(a) Valves averaged from small representative samples.

(b) Determined by leaching at analytical laboratory.

(c) Broken particles from which heavy metals came - based on analytical leaching analysis.

TABLE 15
COMPARISON OF BROKEN PARTICLE DATA (a)

	Crusher		Total		Burner (by difference)	
	Run 38	Run 39	Run 38	Run 39	Run 38	Run 39
U from broken particles, g	8.088	4.268	8.549	7.500	0.461	3.232
Th from broken particles, g	403.39	192.96	568.64	267.77	165.25	74.81
Number of fissile broken particles	2,344,000	371,000	2,478,300	651,500	134,000	280,500
Percent of total fissile particles in feed	3.67	1.40	3.87	2.46	0.20	1.06
Number of fertile broken particles	1,157,000	729,000	1,668,400	984,000	511,400	255,000
Percent of total fertile particles in feed	11.99	5.26	17.29	7.10	5.30	1.84

(a) Results determined by analysis of small representative samples at analytical laboratory and scaled up to correspond to weight of feed entering burner (basis).

to the difference in particle diameters. It would be expected that the smaller of two particles with different diameters but the same SiC layer thickness would be physically stronger. In addition, the relative breakage of the fissile to fertile particles was larger in Run 39, which is probably directly related to the smaller difference in mean particle diameters.

4.1.1.9. Runs 40 and 41

Runs 40 and 41 were performed with fines recycle to investigate the effects of a second O_2 inlet stream on burner operation. The average bed weight for each run was approximately 4.5 kg, and the average burn rates were 33 to 34 g C/min. Eighteen samples of fines were taken during the two runs, giving the data presented in Table 16. Half of the samples were taken while operating with a second O_2 inlet stream into the burner at a location 4 in. above the bottom of the burner vessel (middle of fluidized bed) and at an angle 30° above horizontal. This downward angle was utilized to compensate for the upward deflection caused by the primary fluidizing gas.

From the data of Table 16, there appears to be no significant effect on fines elutriation attributable to use of the second O_2 stream. Samples taken toward the end of each run showed considerably larger rates than those in the beginning due to a larger fines content in the mixed recycle feed to the burner. When the amount of fines in the mixed recycle feed ideally becomes constant (an unlikely event with mixed feed due to problems resulting from hopper design and lack of uniform flow), the fines elutriation rate should become nearly constant. It would seem advantageous to operate with separate feed streams (fines and $<3/16$ -in. fresh feed) such that control of each stream could give the necessary flows to achieve optimum bed burning characteristics and fines elutriation rates.

TABLE 16
FINES DATA - RUNS 40 AND 41

Gas Flows (SLPM)		Burn Rate (g C/min)	Bed Size (kg)	Top of Burner Superficial Velocity (cm/sec)	Fines Rate (g C/min)
Cone Gas	2 nd O ₂				
72.3	-	31.6	4.5	37.2	26.4
75.3	-	32.3(a)	4.0	42.2	71.5
77.8	-	33.0	4.5	41.5	34.0
77.3	-	33.7	4.4	43.7	40.8
79.0	-	33.8	4.5	44.3	38.7
79.0	-	33.6	4.5	43.5	39.4
77.4	-	33.2	4.4	44.0	49.2
77.4	-	33.2	4.4	43.4	38.8
74.5	-	34.0(a)	4.4	42.3	56.0
76.0	8	33.6	4.5	47.4	44.0
72.3	10	32.0	4.5	45.1	38.5
70.0	6	30.8(a)	4.5	42.0	58.6
67.3	8	34.8	4.6	41.8	35.3
65.2	8	34.5	4.4	43.1	44.9
68.9	8	34.9	4.5	44.3	44.8
68.9	8	34.9	4.5	44.6	47.3
65.9	8	33.6(a)	4.4	39.5	47.8
64.8	12	35.6(a)	4.5	43.5	47.6

(a) Large amount of fines in mixed recycle feed (samples taken toward end of run).

Table 17 presents several burner temperature profiles showing the effects of the second O_2 inlet on burner temperature. In all cases, the burning zone was lengthened considerably with the second O_2 inlet in operation. The burn rates were essentially constant, thereby illustrating that the heat production was spread over a larger heat transfer area. One possible advantage of a larger burning zone would be that the size distribution of graphite burned would be more uniform (less preferential) such that bed segregation would be less likely to occur. In addition, heat transfer would likely be enhanced due to a larger concentrated heat source.

4.1.1.10. Runs 43A and 43B

Runs 43A and 43B were performed to acquire more data with which to evaluate the effects of a second O_2 inlet stream. Run 43A was terminated during the initial bed buildup procedure when fresh feed backed up through the recycle auger system causing the augers to bind. As in several previous runs, the problem was caused by the nature of the material pickup point (Ref. 5) of the tapered bottom auger and the inability of this auger to transport feed fast enough. Run 43B was performed with an average burn rate of 37.7 g C/min in a bed varying from 3.7 to 4.7 kg. The second O_2 inlet was used during approximately half the run, with the O_2 flow accounting for approximately 30% of the total O_2 flow at any given time. This represented an increase over Runs 40 and 41 where the second O_2 inlet accounted for only 15% of the total O_2 flow.

Table 18 shows the fines samples taken during Run 43. Again, there appears to be little or no difference in fines elutriation rates between operation with and without secondary O_2 . Fines elutriation is apparently primarily a function of burner superficial velocity (top) and the total weight of fines in the bed. The total weight of fines in the bed that can elutriate will be the net result of fines entering in the feed, leaving in the product, being burned in the bed, and being generated in the bed by

TABLE 17
TEMPERATURE PROFILES - RUN 41

	Gas Flows (SLPM)			Thermocouple Reading (°C)								ΔT Across Bed (°C)
	Cone N ₂	Cone O ₂	2 nd O ₂	1	2	3	4	5	6	7	8	Thermocouples 1-5
Initially	13.7	51.5	8.0	912	901	889	874	855	835	670	470	57
4 min after flow change	21.2	58.0	0	941	925	888	842	781	755	689	482	160
Initially	20.0	59.0	0	916	906	877	840	800	780	670	464	116
4 min after flow change	16.4	52.5	8.0	899	895	890	881	864	838	661	462	35
Initially	19.9	57.5	0	919	906	880	851	816	760	590	433	103
4 min after flow change	15.0	50.9	8.0	921	916	911	904	864	766	584	426	57

TABLE 18
FINES DATA - RUN 43

Gas Flows (SLPM)		Burn Rate (g C/min)	Recycle Auger (rpm)	Bed Size (kg)	Top of Burner Superficial Velocity (cm/sec)	Fines Rate (g C/min)
Cone Gas	2 nd O ₂					
73.0	0	34.8	4.5	4.3	41.8	34.6
73.0	0	34.9	4.5	4.7	42.2	34.8
67.0	20.0	39.2	4.0	4.6	52.5	46.0
67.5	19.5	36.8(a)	9.5	3.9	50.9	57.5
67.5	19.5	36.8(a)	10.0	4.0	51.5	55.0
67.5	19.5	36.6(a)	16.0	3.7	53.5	75.0
74.0	0	34.3(a)	18.0	4.3	43.5	70.5

(a) Large amount of fines in mixed recycle feed (samples taken toward latter part of run).

burning of large pieces of graphite. The trend of the fines rates shown in Table 18, as well as in other runs, is to generally increase with time since the fines content of the mixed recycle feed and the required mass flow into the burner also increase. Thus, it can be shown that when the weight of fines in the feed becomes relatively large, the weight of fines in the feed is usually the most dominant factor contributing to the fines elutriation rate. The amount of fines burning and fines generation in the bed, and the operating variables on which they depend, are not clearly understood at this time. In any event, their relative effect on the fines elutriation rate would seem to decrease as the weight of fines in the feed increases. It appears that a system utilizing separate feed streams of fresh feed and recycled fines would be better suited for investigating fines elutriation characteristics.

4.1.1.11. Run 42

Run 42 was performed using CO_2 as the cone fluidizing gas diluent instead of N_2 . The objectives of Run 42 were to determine the effects of using CO_2 on burner operation including the CO/CO_2 off-gas ratio, fines elutriation, and burner control. The average burn rate was 32.8 g C/min in a bed varying from 3.4 to 4.1 kg. The off-gas analyses shown in Fig. 14 include both the actual CO_2 concentration and the molar concentration of CO_2 produced by the graphite combustion. This latter concentration is obtained by subtracting out the diluent concentration. Table 19 is a comparison of average ratios of CO/CO_2 produced by combustion for several runs made under comparable operating conditions. It can be seen that the values for these runs agree reasonably well, thereby indicating little or no change in reaction characteristics as a result of using CO_2 as the diluent. The fact that there was no significant increase in the CO/CO_2 off-gas ratio demonstrates that use of a diluent (whether it be N_2 or CO_2) will affect the combustion reaction only to the extent that the partial pressure of oxygen is reduced and will not affect the CO/CO_2 ratio (corrected ratio in the case of CO_2 diluent).

Fig. 14. Off-gas analysis, Run 42

TABLE 19
COMPARISON OF AVERAGE OFF-GAS CO/CO₂ RATIOS

Temperature (°C)	Average CO/CO ₂ Ratio		
	Run 42 (a)	Run 35	Run 43 (no 2 nd O ₂)
880-900	0.180	0.192	0.199
900-920	0.203	0.193	0.195
880-920	0.193	0.192	0.197
920-940	0.213	-	0.218
940-960	0.230	-	-

(a) Corrected to account for cone CO₂.

Table 20 gives the fines elutriation data from Run 42. As in Runs 40, 41, and 43, the data indicate that the most dominant factor contributing to fines elutriation rates is the weight of fines in the recycle feed at any given time. The actual values of the fines rates are consistent with previous runs, indicating that no change in elutriation characteristics resulted from the use of CO_2 diluent. The only apparent changes in burner operation attributable to the use of CO_2 diluent were caused by the higher molar heat capacity of CO_2 , which makes burner control more sensitive to gas control. It was noted that the temperature transients occurred much faster. In addition, the initial bed ignition seemed to be affected by the use of fluidizing CO_2 in that it took somewhat longer than usual for startup. This could be attributed to the endothermic CO_2 reduction, which would be more significant with CO_2 diluent particularly during startup. However, this was only a minor inconvenience and not a significant problem.

4.1.1.12. Runs 44 and 45

The objectives of Runs 44 and 45 were to acquire heat transfer data while using an increased supply of clamshell cooling air along with higher burn rates than previously used. Experimental overall heat transfer coefficients were obtained for operation with and without secondary oxygen. Run 44 was performed without secondary oxygen at an average burn rate of 42.3 g C/min in a bed varying from 4.0 to 4.5 kg. The maximum attainable burn rate was approximately 57 g C/min. The CO/CO_2 off-gas ratio was lower than usual in this run, which can be attributed to the low bed carbon content during most of the run. With low bed carbon content, the CO_2 -C reduction reaction was decreased. In addition, with the increased amount of fluidizing O_2 required to achieve a maximum burn rate (fluidizing gas was 100% O_2 in order to minimize fluidization velocity), the velocity was quite high, thereby tending to spread out the volume of the burning zone and causing slower O_2 utilization. Subsequently, CO formed by reaction in the bed would be more likely to undergo homogeneous oxidation to CO_2 before

TABLE 20
FINES ELUTRIATION DATA - RUN 42

Cone Gas Flow (SLPM)	Burn Rate (g C/min)	Recycle Auger (rpm)	Bed Size (kg)	Top of Burner Superficial Velocity (cm/sec)	Fines Rate (g C/min)
67.2	31.2	4.0	4.0	33.9	18.8
69.9	30.9	5.0	3.7	36.7	35.1
68.5	30.0	7.5	3.7	38.3	43.2
69.1	31.5 ^(a)	11.0	4.0	40.1	62.8
69.1	31.3 ^(a)	11.5	3.8	39.3	66.0
66.8	32.7 ^(a)	13.5	3.9	39.6	59.7
65.6	32.7 ^(a)	11.5	3.9	38.2	73.5
68.1	31.1 ^(a)	16.5	3.8	39.1	77.3
65.7	32.4 ^(a)	19.0	4.0	37.7	77.5

^(a) Large amount of fines in mixed recycle feed (samples taken toward latter part of run).

leaving the burner vessel. This, together with the less significant decrease in CO_2 reduction, resulted in a decrease in the CO/CO_2 off-gas ratio.

Table 21 shows the fines elutriation data of Run 44. As in previous runs, the fines elutriation rates were approximately 2 to 3 times greater than the corresponding burn rate. Unlike previous runs, the total feed rate was so high that a steady-state condition in the mixed feed size distribution was approached, and thus the fines elutriations rates were essentially constant.

Table 22 shows the experimental heat transfer data of Run 44, and Fig. 15 shows the cooling air flow calibrations for each clamshell at various control valve settings. Overall heat transfer coefficients ($\bar{U}$) were obtained for each clamshell and are plotted in Fig. 16 along with data from Runs 38 and 39 (less cooling air). The data from Runs 38 and 39 were obtained with a laminar flow of cooling air ($\text{Re} < 2000$), while the data of Run 44 were obtained with cooling air flows in the transition or turbulent flow range ($2,500 < \text{Re} < 16,000$). Subsequent calculations showed that the heat transfer resistance outside the burner vessel (convection along the inner clamshell and outer burner surfaces) was the controlling heat transfer resistance for fully turbulent cooling air flow. As the cooling air flow Reynolds number increases, the outer burner heat transfer resistance becomes more and more controlling.

Run 45 was performed with secondary oxygen at an average burn rate of 45.9 g C/min in a bed varying from 4.0 to 5.0 kg. As in Run 44, the maximum attainable burn rate while using maximum cooling air and operating at less than 1000°C was approximately 57 g C/min. Heat transfer data were obtained while using cooling air flows with Reynolds numbers of approximately 2,500 to 16,000 (40 to 100% control setting) and are shown in Table 23. Table 24 compares data from Runs 44 and 45. It can be seen that

TABLE 21
FINES ELUTRIATION DATA - RUN 44

Cone Gas Flow (SLPM)	Burn Rate (g C/min)	Recycle Auger (rpm) (a)	Bed Size (kg)	Top of Burner Superfical Velocity (cm/sec)	Fines Rate (g C/min)
86.5	49.5	23.0	4.3	54.6	132.6
86.5	49.5	26.5	4.2	53.5	121.1
91.0	51.9	26.5	4.4	53.1	116.7
98.0	56.5	26.5	4.3	58.2	120.0
98.0	56.5	26.5	4.3	57.1	122.4

(a) Approximately 50 to 60 wt % fines in mixed recycle feed.

TABLE 22
HEAT TRANSFER DATA - RUN 44

Cooling Air Setting (%)	Clamshell	Clamshell Air Flow (at 25°C) (ft ³ /min)	Clamshell Heat Removal, Q		ΔT_{1n} (°F)	$\bar{U} \left(\bar{U} = \frac{Q}{A \Delta T_{1n}} \right)$ (Btu/hr-ft ² -°F)	Total Heat By Combustion (Δ hr x Btu/hr)
			Btu/hr	%			
40	1 (bottom)	20.9	14,643	36.3	1,193	10.4	65,000
	2	19.2	10,778	26.8	1,220	7.5	
	3	25.3	9,310	23.1	1,111	7.1	
	4 (top)	26.2	5,556	13.8	812	3.9	
40	1	20.9	14,513	39.6	1,288	9.6	71,000
	2	19.2	10,299	28.1	1,275	6.9	
	3	25.3	6,785	18.6	1,119	5.2	
	4	26.2	5,011	13.7	807	3.5	
50	1	39.3	20,998	38.4	1,299	13.7	79,000
	2	34.9	15,672	28.6	1,272	10.5	
	3	43.6	9,880	18.1	1,171	7.2	
	4	43.6	8,158	14.9	897	5.2	
50	1	39.3	20,508	38.9	1,251	13.9	71,000
	2	34.9	15,092	28.6	1,237	10.4	
	3	43.6	9,064	17.2	1,143	6.7	
	4	43.6	8,067	15.3	870	5.3	
55	1	52.5	24,776	40.8	1,331	15.8	79,000
	2	47.0	18,077	29.8	1,281	12.0	
	3	54.0	8,981	14.8	1,152	6.6	
	4	54.8	8,886	14.6	872	5.8	
55	1	52.5	24,776	41.7	1,355	15.5	84,000
	2	47.0	17,491	29.4	1,303	11.4	
	3	54.0	8,644	14.5	1,162	6.3	
	4	54.8	8,545	14.4	884	5.5	

TABLE 22 (Continued)

Cooling Air Setting (%)	Clamshell	Clamshell Air Flow (at 25°C) (ft ³ /min)	Clamshell Heat Removal, Q		ΔT_{1n} (°F)	$\bar{U}$ $\left(\bar{U} = \frac{Q}{A \Delta T_{1n}} \right)$ (Btu/hr-ft ² -°F)	Total Heat By Combustion (Δ hr x Btu/hr)
			Btu/hr	%			
60	1	74.2	31,007	45.6	1,382	19.1	84,000
	2	65.5	20,699	30.4	1,301	13.5	
	3	65.5	8,443	12.4	1,122	6.4	
	4	65.5	7,898	11.6	839	5.3	
70	1	110.9	41,500	50.9	1,390	25.3	88,000
	2	94.5	22,200	27.2	1,278	14.8	
	3	91.1	10,227	12.6	1,069	8.1	
	4	82.9	7,583	9.3	813	5.3	
100	1	119.7	42,547	50.7	1,374	26.3	93,000
	2	103.2	23,391	27.9	1,269	15.7	
	3	97.5	10,541	12.5	1,051	8.5	
	4	90.0	7,484	8.9	793	5.3	
100	1	119.7	42,547	48.8	1,382	26.1	93,000
	2	103.2	25,532	29.2	1,293	16.8	
	3	97.5	11,351	13.0	1,083	8.9	
	4	90.0	7,859	9.0	815	5.5	
100	1	119.7	46,038	49.5	1,444	27.1	98,000
	2	103.2	27,034	29.0	1,325	17.3	
	3	97.5	11,757	12.6	1,103	9.1	
	4	90.0	8,233	8.9	833	5.6	
100	1	119.7	48,029	52.0	1,472	27.7	98,000
	2	103.2	25,532	27.6	1,322	16.4	
	3	97.5	11,351	12.3	1,067	9.0	
	4	90.0	7,484	8.1	799	5.3

Fig. 15. Cooling air calibrations, Run 44, 10-cm primary burner

Fig. 16. Heat transfer calculations, Run 44, 10-cm primary burner

TABLE 23
HEAT TRANSFER DATA - RUN 45

Cooling Air Setting (%)	Clamshell	Clamshell Air Flow (at 25°C) (ft ³ /min)	Clamshell Heat Removal, Q		ΔT_{1n} (°F)	$\bar{U} \left(\bar{U} = \frac{Q}{A \Delta T_{1n}} \right)$ (Btu/hr-ft ² -°F)	Total Heat By Combustion (Δ hr x Btu/hr)
			Btu/hr	%			
40	1 (bottom)	20.9	13,904	35.2	1,185	10.0	64,000
	2	19.2	9,979	25.3	1,193	7.1	
	3	25.3	10,415	26.4	1,122	7.9	
	4	26.2	5,175	13.1	741	4.0	
55	1	52.5	21,502	39.7	1,316	13.9	75,000
	2	47.0	16,904	31.2	1,289	11.1	
	3	54.0	9,543	17.6	1,111	7.3	
	4	54.8	6,266	11.5	789	4.5	
60	1	74.2	29,927	42.5	1,360	18.7	80,000
	2	65.5	21,379	30.3	1,327	13.7	
	3	65.5	9,941	14.1	1,180	7.2	
	4	65.5	9,260	13.1	919	5.7	
70	1	110.9	40,348	46.5	1,387	24.7	89,000
	2	94.5	25,344	29.2	1,317	16.3	
	3	91.1	11,553	13.3	1,131	8.7	
	4	82.9	9,479	11.0	881	6.1	
70	1	110.9	41,732	46.8	1,393	25.4	93,000
	2	94.5	26,326	29.5	1,341	16.7	
	3	91.1	11,743	13.2	1,150	8.7	
	4	82.9	9,307	10.5	885	6.0	
100	1	119.7	45,292	48.5	1,430	26.9	98,000
	2	103.2	27,034	28.9	1,340	17.1	
	3	97.5	11,959	12.8	1,127	9.0	
	4	90.0	9,169	9.8	869	6.0	
100	1	119.7	47,531	49.7	1,473	27.4	98,000
	2	103.2	27,034	28.2	1,359	16.9	
	3	97.5	11,757	12.3	1,133	8.8	
	4	90.0	9,356	9.8	876	6.0	

TABLE 24
COMPARISON OF RUNS 44 AND 45

Cooling Air Setting (%)	Total Heat By Combustion (Δ hr x Btu/hr)		Clamshell	Clamshell Heat Removal, Q				$\bar{U} \left(\bar{U} = \frac{Q}{A\Delta T_{1n}} \right)$ (Btu/hr-ft ² -°F)	
				Run 44		Run 45		Run 44	Run 45
	Run 44	Run 45		Btu/hr	%	Btu/hr	%		
40	65,000	64,000	1 (bottom)	14,643	36.3	13,904	35.3	10.4	10.0
			2	10,778	26.8	9,979	25.3	7.5	7.1
			3	9,310	23.1	10,415	26.4	7.1	7.9
			4 (top)	5,556	13.8	5,175	13.1	3.9	4.0
55	79,000	75,000	1	24,776	40.8	21,502	39.7	15.8	13.9
			2	18,077	29.8	16,904	31.2	12.0	11.1
			3	8,981	14.8	9,543	17.6	6.6	7.3
			4	8,886	14.6	6,266	11.5	5.8	4.5
60	84,000	80,000	1	31,007	45.6	29,927	42.5	19.1	18.7
			2	20,699	30.4	21,379	30.3	13.5	13.7
			3	8,443	12.4	9,941	14.1	6.4	7.2
			4	7,898	11.6	9,260	13.1	5.3	5.7
70	88,000	89,000	1	41,500	50.9	40,348	46.5	25.3	24.7
			2	22,200	27.2	25,344	29.2	14.8	16.3
			3	10,227	12.6	11,553	13.3	8.1	8.7
			4	7,583	9.3	9,479	11.0	5.3	6.1
100	98,000	98,000	1	48,029	52.0	47,531	49.7	27.7	27.4
			2	25,532	27.6	27,034	28.2	16.4	16.9
			3	11,351	12.3	11,757	12.3	9.0	8.8
			4	7,484	8.1	9,356	9.8	5.3	6.0

the most significant effect of operating with secondary oxygen (20 to 25% of total O_2 flow) was to lengthen the burning zone such that relatively more heat was removed in the upper section of the burner and less in the lower section. However, the differences were quite small (several percent) and would be even smaller in larger diameter burners.

Due to continuing problems with the recycle auger feed system (Refs. 4,5), a new untapered auger will be installed (lower auger), the speed of which will be increased relative to the upper auger. This should improve the efficiency of material transport at the pickup point located at the bottom of the vertical bellows connection. Problems have occurred when failure of the lower auger to transport feed as fast as it receives it from the upper auger has caused the feed lines to back up.

4.1.2. 20-cm Primary Burner

Fabrication and assembly of the burner system is complete, as shown in Fig. 17. Shake-down runs using graphite feed are in progress to acquaint personnel with operational procedures and to permit detailed calibration and testing of equipment and instrumentation.

Late delivery of major venturi components required in the pneumatic fines recycle feeding system delayed testing of the fines injection system. These parts are now assembled and fines feeding tests are in progress with a bench system simulating burner back pressure.

The initial burner runs will characterize fines elutriation with top and bottom fresh feed without recycle of fines. Concurrent with these runs, the pneumatic fines feeder will be bench optimized and calibrated prior to inclusion in the operating burner process.

The recycled fines stream will be premixed with oxygen just upstream of the burner (Fig. 17) in the first fines burning runs. After these

Fig. 17. 20-cm primary burner

tests, a vortex inducer spool will be incorporated in the fines-oxygen feed line (Fig. 18). The location of the fines injection to the burner, the premixing with oxygen, and the vortex induction are measures to maximize fines mixing and residence time in the oxygen-rich combustion zone of the burner. The vortex concept is based on the supposition that particle - gas stream contact (turbulent diffusion) is less effective and hence becomes the controlling factor as the particle size becomes finer and the relative gas stream to particle velocity decreases (Ref. 8). The bottom burner location of the fines injection stream is also considered critical; the larger particles in the lower bed will burn faster than the smaller recycled fine particles unless the fines injector causes a high degree of mixing.

A recommended design for the prototype primary burner gas distribution has been developed based on the postulated fines burning criteria and the restraints of hot cell safety and remote operation. This distributor will be tested on the revamped 20-cm primary burner after fabrication is complete and background data have been generated in the present 8-in. system with fines recycle and vortex induction.

The distributor concept is shown in Fig. 19. This concept incorporates a perforated conical distributor shrouded in a gas plenum. The perforated cone was chosen as the most attractive for primary burning as it allows tangential injection of the fines stream, yielding the desired vortex in the oxygen-rich burner zone. Further, the sloping cone walls provide the minimum solids stagnation potential, and hence less chance of distributor burn-through. The point of product removal at the inverted cone apex is the most favorable in terms of product accountability in a larger burner and, with the properly sized product take-off line and metered gas rates, product may be excluded from the removal line during burning and possibly be separated by gas classification during product take-off periods (Ref. 9).

Fig. 18. 20-cm burner with vortex inducer spool

Fig. 19. Perforated cone and plenum gas distributor

The bulk of the fluidization-combustion gas will be routed to the cone plenum and then through the perforated cone. The recycled fines stream will be carried by additional gas, the exact quantities of which will be determined by current pneumatic fines transport testing. The remainder of the process gas will be introduced through the cone inverted apex at rates metered on the basis of the solids terminal velocities.

Prior to actual burning runs, the distributor will be attached to an existing 41-cm plexiglass column to observe gross effects on fluidization with different gas and fines injector inlets.

4.1.3. Prototype Primary Burner

The prototype conceptual design has been completed based upon operating experience in the present pilot plant. The experimental program on the smaller burners is being adjusted to allow verification of some design concepts that have not been demonstrated on smaller systems with crushed graphite and HTGR fuel particles. However, none of the concepts are novel to fluidized bed combustor technology.

4.1.3.1. System Arrangement

A pneumatic transfer system will be used between unit operations. The layout of equipment will be similar to the existing 20-cm primary burner. Layout drawings are included in Section 7, Systems Design (Fig. 51).

4.1.3.2. Top Fresh Feed

The reference prototype design uses top fresh feed of crushed fuel elements. Based on pilot plant experience with mixed feed, it is better to feed fresh feed separately from recycled fines. Also, top feed eliminates alignment problems between the feed hopper and burner vessel when utilizing bottom feed. These alignment problems occur during thermal expansion of the burner vessel.

At present it is felt that a positive seal (air-lock) is needed to prevent process upsets in the burner as a result of anticipated pressure and feed rate surges in the pneumatic transfer system. This seal would also prevent the release of burner process off-gas, which will contain a relatively high concentration of fission products, into the pneumatic transfer off-gas system.

It may be possible to utilize a pneumatically assisted gravity feed system. This system has been developed (see Section 4.2.1, "10-cm Secondary Burner") and tested utilizing primary burner feed (Ref. 4). This alternate feed method has the advantage of possessing no moving parts. The system will be further tested on the 20-cm primary burner.

4.1.3.3. Pneumatic Fines Recycle and Gas Distribution

The ability to burn recycled fines is dependent on the method of fines injection and the design of the gas distributor. The combination of a perforated conical distributor with tangential fines injection and a perforated cone has been successfully utilized on coal combustion units, with reported high fines burning efficiencies (Ref. 10). Although coal combustors are not exact duplicates of the primary burner, both the fresh feed and the recycled fines have comparable size distributions and densities for the two systems. The fresh feed for coal combustors is <1/4-in. coal mixed with limestone; the primary burner feed is <3/16-in. graphite mixed with SiC coated HTGR fuel particles.

Because confirmatory backup process data on the pilot-scale units will not be available until the prototype design is completed (see Section 4.1.2, "20-cm Primary Burner"), the gas distributor plenum (Fig. 20) is designed to allow incorporation of a flat perforated plate without major modification of the gas distributor plenum. The flat perforated gas distributor plate is the backup design; confirmation of this design is being obtained at ACC-ICPP.

4.1.3.4. Induction Heating System

Preliminary design of the induction heating system for startup has been completed. The design includes an induction coil inside the air cooling jacket (Fig. 21). A susceptor will be used as the heating source, instead of direct susception of the vessel wall. A 1/2-in. annular gap for cooling air will be maintained between the vessel wall and the susceptor. During startup, the vessel wall and bed materials will be heated by radiant heat transfer from the susceptor across the annular gap. To prevent excessive heat loss to the coil, a rigid insulation will be spaced between the susceptor and the coil. The induction coil is held in place with transite ribs positioned at intervals. The design criteria used in arriving at this design are discussed in the following sections.

4.1.3.4.1. Overall Efficiency of the Induction Heating System. The overall efficiency consists of the electrical efficiency of the coil and the thermal efficiency against heat loss through the insulation. The electrical coil loss can be estimated if the coil physical dimensions (for example, insulation thickness and gap between work pieces) are fixed. The thermal loss is a function of the thermal conductivity and thickness of the insulation and can be determined from a startup temperature transient analysis.

Two types of rigid insulations (from Glasrock Products, Inc.) are being considered:

$$\text{Masrock} \left(k = 7.5 \frac{\text{Btu}}{\text{hr-ft}^2/\text{in.}-^\circ\text{F}} \right)$$
$$\text{Foam 50} \left(k = 1.8 \frac{\text{Btu}}{\text{hr-ft}^2/\text{in.}-^\circ\text{F}} \right) .$$

SCALE 1/1

- 1 REMOVE ALL BURRS AND SHARP EDGES.

2 DIMENSIONS AND TOLERANCES PER ANSI Y14.5.

3. WELD BY MANUAL ARC, MIG OR TIG PROCESS.

4. LIQUID PENETRANT INSPECT WELDS PER
ASTM-E165.

REQD/ASSEMBLY				

כח

Fig. 21. Prototype 40-cm burner remote flange and heater

Masrock is not as good an insulator as Foam 50, but being harder can withstand high-velocity air erosion without any protection. On the other hand, Foam-50 requires a susceptor for protection against air erosion (see Fig. 22).

The total energy loss to the coil cooling water as a function of insulation thickness is shown for both insulations in Fig. 23. This figure shows a distinct advantage for the Foam-50 insulation with a susceptor.

Further consideration of pressure drop and heat dissipation by a closed-loop coil cooling water system limits the choice of insulation thickness to ~ 2 in. and the outside diameter of the coil tubing to 1 in. (Fig. 24).

4.1.3.4.2. Startup Temperature Transient Analysis. With the selected 2-in. insulation thickness, a startup transient was analyzed for two modes of startup operation:

1. "Whole bed" startup from ambient (Fig. 25).
2. Preheating of vessel wall to 900°C followed by "rapid dump" of bed material (Fig. 26).

The transients show that a 250 kW motor-generator set at 1 kHz is sufficient to start up a bed of fresh feed (three fuel elements) to stable ignition (at $\sim 850^{\circ}\text{C}$) in approximately 100 min. The mode of startup has only a slight effect on the total startup time. However, the whole bed startup from ambient has a lower rate of wall temperature rise, which is a major concern for the remote clamp design.

The susceptor is limited to a maximum temperature of 1100°C , and the vessel wall temperature has a maximum limit of 900°C .

Fig. 22. Masrock and Foam-50 insulation

Fig. 23. Total electrical and thermal loss to coil cooling water as a function of insulation thickness

Fig. 24. Induction coil parameters as a function of insulation thickness (prototype 40-cm primary burner with 1/8-in. susceptor plate)

Fig. 25. Temperatures for whole bed startup from ambient

Fig. 26. Temperatures for "rapid dump" startup with tube initially at 900°C

4.1.3.4.3. Addition of Susceptor. The presence of a susceptor has added an extra safety feature to the design. The susceptor acts as a secondary containment for the bed materials in the event of a vessel wall burn-through. Susceptor heating will also provide more uniform heating of the vessel since possible impurities in the vessel wall or miscentering of the vessel around the coil can produce local hot spots in the vessel if direct susception is employed. Also, the option to add a finned burner tube for added heat transfer area is available with the susceptor design concept.

4.1.3.5. Gas Cooling of Vessel

Two air cooling clamshells are used for external cooling of the vessel wall during steady-state burning at a maximum burn rate of 800 g C/min (Fig. 27). The lower 6-ft clamshell also houses the induction heating coil and susceptor. A blower is used to force cooling air through the 1/2-in. annular gap between the vessel wall and the clamshell for each section.

Preliminary design indicates that the major resistance to heat transfer is the internal vessel wall film coefficient. The overall heat transfer coefficient used in the present design is $17 \text{ Btu/hr-ft}^2\text{-}^\circ\text{F}$. This value utilizes experimental data on the inside wall coefficient and an outside coefficient corrected for the design cooling gas rate.

4.1.3.6. Entrained Fines Separation From Process Off-Gas

A cyclone [Fisher-Klosterman, Inc., Model XQ-5(S), 12-gauge 316 SS] has been sized to separate entrained fines. Vendor fabrication of the cyclone is under way. A pulsed blow-back filter assembly with a cam timer will be used to remove any final trace of off-gas particulates before the off-gas is discharged for treatment.

Fig. 27. 20-cm primary burner assembly

4.1.3.7. Fully Remote High-Temperature Flange

The remote high-temperature flange is being designed by the vendor (Gray Tool Co.).

4.1.3.8. Main Vessel and Flanges

The main vessel assembly drawings have been completed (Fig. 28). The vessel will be made of Hastelloy-X except for the upper vessel cap assembly, which will be 316 SS. Of the three flanges assemblies, only the middle flange has the fully remote clamp. The other two flanges are semi-remote clamps (one-bolt swing-away). This portion of the prototype burner system will be designed and fabricated in accordance with the ASME Boiler and Pressure Code, Section VIII, Division 1. This requires the use of Hastelloy-X as the material of construction for those portions of the vessel where the metal temperatures will be above 650°C.

A semi-remote maintenance philosophy will be incorporated in the design as discussed in Section 7.

4.2. SECONDARY BURNER

4.2.1. 10-cm Secondary Burner

As recommended previously (Ref. 1), an induction-heated Hastelloy-X burner tube has been installed and tested. Three experimental runs (37, 38, and 39) were made using the new induction-heated burner system with two different coil configurations (one with insulation and the other without). Addition of partial beds for startup was found to speed ignition time and is now an operating procedure. Increased filter blow-back capacity yielded low filter pressure drops and will be explored further. After initial problems, pneumatic product transport became operable with attainment of complete distributor plate clean-off.

- NOTES
1. DO NOT SCALE DRAWING.
 2. REQUIRED FOR PRESSURE TEST ONLY.

① BURNER VESSEL ASSEMBLY

DETAIL A
CONE ATTACHMENT

ITEM	PART NO.	DESCRIPTION	MATL
1	28	GRAYLOC 170 SEAL RING	HASTELLOY "X"
2	27	GRAYLOC 140 SEAL RING	HASTELLOY "X"
1	26	GRAYLOC 52 SEAL RING	A286 SS
2	25	GRAYLOC 40 SEAL RING	A286 SS
2	24	GRAYLOC 11 SEAL RING	A286 SS
1	23	GRAYLOC 7 SEAL RING	A286 SS
2	21	GRAYLOC 4" BLIND HUB	316 SS
20	GR-11	GRAYLOC 1" BLIND HUB	316 SS
19			
18			
17			
1	16	GRAYLOC 5" CLAMP	CF8M
1	15	GRAYLOC 4" CLAMP	CF8M
3	14	GRAYLOC 1" CLAMP	CF8M
1	13	2410013 18" QUICK DISCONNECT CLAMP	HASTELLOY "X"
1	12	2410012 14" REMOTE CLAMP	HASTELLOY "X"
1	11	2410011 14" QUICK DISCONNECT CLAMP	HASTELLOY "X"
10	2410014	THERMOWELL PROBE	
9			
8	2410010	DISTRIBUTION CONE	
7	2410008	LOWER CAP ASS'Y.	
6	2410006	SPOOL ASS'Y.	
5	2410005	BURNER TUBE ASS'Y.	
4			
3			
2	2410004	THERMOCOUPLE FLANGE	
1	2410003	UPPER CAP ASS'Y	
-1		BURNER VESSEL ASSEMBLY	
REQD/ASSEMBLY	LIST OF MATERIAL		

Fig. 28. 20-cm primary burner vessel assembly

4.2.1.1. Run 37

In Run 37, 20 kg of crushed TRISO fertile particles were combusted to 0.8% burnable carbon content using the induction-heated Hastelloy-X 10-cm secondary burner. Increased filter blow-back capacity reduced the peak off-gas filter pressure drop by over 40%. Product removal via the pneumatic transport system after completion of the burning run resulted in choking flow in vertical sections due to high solids-to-air ratios.

A 0.014-in. nominal gap double-roll crusher was used to break the TRISO fertile particles used as feed. This material contained 64.6 wt % ThC_2 , 17.9 wt % C, and 17.5 wt % SiC.

The tube was first heated to 900°C by the induction heater. The gravity-pneumatic feeder was then used to feed the crushed particles. A flow of 80 liters/min of N_2 was used to fluidize the bed being added. The 20-kg bed was added in 2 min, with a continuous N_2 activation flow through the feeder of 25 liters/min. The bed and tube came to an initial equilibrium temperature of $\sim 400^\circ\text{C}$ and were then heated via induction to 600°C over a 20-min period.

When the bed reached 600°C, the fluidizing flow was changed to 40 liters/min O_2 and 40 liters/min N_2 , which gave immediate combustion and subsequent temperature increases with no O_2 detected in the off-gas during ignition. The bed was brought to 900°C using flows as shown in Fig. 29.

Cooling air was supplied to the filter cooling jackets but not to the burner clamshells because there was sufficient heat removal via the induction coil cooling water for the burn rate attained.

From the axial temperature distribution, it is apparent that the bed was consistently slugging into the filter chamber, yielding 800°C temperatures in the filter vicinity. The bed size was decreased by 10% in Run 38 to reduce the filter temperatures.

Fig. 29. Gas flow, Run 37, 10-cm secondary burner

The two filters were alternately blown-back every 30 sec with 10 liters of recycled off-gas per filter. The blow-back gas was supplied at 35 psig. The filter ΔP rose quickly to 25 in. H_2O (as compared to 45 in. H_2O in previous runs) and gradually fell during the run to 15 in. H_2O . Fines buildup on the filters was rapid between blow-back pulses, with increases of up to 5 in. H_2O between each pulse at the start of the run and about 0.5 in. H_2O between blow-back pulses near the end of the run. The higher buildup rate at the start of the run is attributed to the presence of inner pyrolytic and buffer carbon coatings, which are eliminated via oxidation during the run.

Pressure fluctuations due to slugging were observed to be ± 0.2 psi at the frequency of ~ 2 /sec. This is less than previous runs and indicates a more smoothly fluidized bed.

The burner expanded axially (~ 1.1 in.), which is consistent with data supplied by the tube manufacturer. Burner alignment mechanisms performed as expected, and no horizontal tube displacement was observed.

When CO_2 levels in the off-gas began dropping, indicating carbon depletion, the flows were dropped as shown in Fig. 29 and the induction heater was activated. Remote vibration of the filter chamber, using a pneumatic vibrator, aided in returning fines to the bed.

The run was terminated after the burn rate dropped to 0.2 g/min. The bed was fluidized with N_2 at 100 liters/min and cooled to $\sim 200^\circ C$ in 10 min.

The pneumatic transport system was then activated and the product removal valve was opened. This resulted in moving product through the horizontal portion of the transport tube and 3 ft up the vertical section, where it stopped, leaving ~ 4 kg in the burner. The tubes were then

manually emptied, and the 4 kg remaining in the burner were successfully transported pneumatically to the product hopper on the upper deck. A thin layer (1/4-in.) of material (120 g) was left on the distributor plate.

The reason for the initial transport tube blockage was that the flow of product through the product valve, which was opened maximally, was too rapid. To correct this condition, an initial opening stop of 1/8 in. was installed on the actuator, which will considerably restrict flow out of the valve during the bulk of the product removal step. The valve will then be fully opened to remove the remaining heel.

Analysis of the product showed it contained 12,070 g ThO_2 , 128 g C, and 3,810 g SiC. The product size distribution is shown in Fig. 30.

4.2.1.2. Run 38

In Run 38, the induction-heated 10-cm secondary burner was used to oxidize 18 kg of crushed TRISO fertile particles to 1.0% burnable carbon content. A partial bed was added initially to decrease ignition time; the remainder was added when the bed was at operating temperature. The product was pneumatically transported to an upper deck hopper.

A double-roll crusher was used to process the TRISO fertile particles to yield burner feed.

After induction heating the tube to 900°C, 8 kg of crushed feed were added and induction heated to 650°C with 80 liters/min of N_2 used for fluidization. The flow was changed to 40 liters/min of O_2 and 40 liters/min of N_2 for ignition and was then brought to 145 liters/min of O_2 and 20 liters/min of N_2 to achieve a 900°C bed temperature. The remaining 10 kg of crushed feed were then added in 1-kg portions.

Fig. 30. Product size distribution, Run 37, 10-cm secondary burner

The run progressed with the gas flows held constant until the temperature began dropping off due to insufficient carbon content in the bed. At this time, the flows were dropped to 60 liters/min of O_2 and 40 liters/min of N_2 , while the induction heater was reactivated to hold the bed temperature above $800^\circ C$. When the burn rate dropped below 0.3 g/min, combustion was terminated by switching to 100 liters/min of N_2 flow.

After the bed was cooled to $300^\circ C$, the product removal valve was opened 1/8 in. to allow material to be moved to the upper deck by the pneumatic transport system. After 5 min, 12,896 g of product had been collected in the upper deck hopper. The next 10 min brought 1,445 g up to the top deck. The transport system was then shut off and the run was terminated.

Disassembly of the burner revealed a thin layer of product (143 g) on the distributor plate, which also occurred in Run 37.

The product contained 1.0 wt % C and had a bulk density of 2.4 g/cm^3 and a tap density of 3.0 g/cm^3 . The size distribution is shown in Fig. 31.

4.2.1.3. Run 39

In Run 39, 18 kg of crushed TRISO fertile particles were burned to 0.6% carbon content in the secondary burner using a modified induction heating coil. The product was pneumatically transported to a product hopper. Use of a distributor plate sweep yielded complete plate clean-off.

A double-roll crusher was used to prepare the TRISO fertile particles for oxidation. Size distributions of the crushed and uncrushed feed are shown in Fig. 32. The crushed feed had an angle of repose of 27° , a bulk density of 1.8 g/cm^3 , and a tap density of 2.2 g/cm^3 .

Fig. 31. Product size distribution, Run 38, 10-cm secondary burner

Fig. 32. Feed size distribution, Run 39, 10-cm secondary burner

The burner was run as described in Ref. 11. Using the new induction coil, the tube was preheated to 800°C. The partial bed was then introduced, equilibrated at 550°C, and ignited by introduction of oxygen. The bed temperature was brought to 900°C and the remainder of the batch was then added. The burn rate was increased to 88 g/min carbon displacement with heat removal via water cooling coils and air jacket cooling. This rate continued for 1 hr, followed by 20 min of reduced flow during the final burnout. The blow-back rate was 10% of the process flow, yielding a peak filter pressure drop of 35 in. H₂O.

After shutdown, the bed was cooled to 300°C over a 10-min period, followed by pneumatic transport of the product to an upper deck hopper. The valve was opened maximally, 1/8 in. after run 37, in order to remove the bulk of the product (80% was removed in 5 min). The distributor plate sweep was then used to remove the heel over a 10-min period. Subsequent disassembly of the burner revealed a clean distributor plate with no heel whatsoever.

The product contained 0.6% burnable carbon. It had an angle of repose of 33°, a bulk density of 2.4 g/cm³, and a tap density of 3.0 g/cm³. The size distribution of the product is shown in Fig. 33.

4.2.1.4. Gravity-Pneumatic Feeder for Fluid Bed Burners

A gravity-pneumatic feeder has been developed that is capable of both primary and secondary burner top feeding. Excellent flow control, high throughputs, lack of moving parts, and no blockages are features inherent in the feeder design.

A gravity-pneumatic feeding system for fluid bed burners has been studied and used for 2-1/2 yr. The original work was done to develop an intermittent feeder for the 20-cm primary burner. A 1-in.-diameter or 3/4-

Fig. 33. Product size distribution, Run 39, 10-cm secondary burner

in.-diameter feeder proved to be optimum for primary burner feed, and could be used by pulsing in small amounts of material intermittently. This burner was deactivated shortly thereafter and is just now being renovated.

This feeder operates by disturbing the angle of repose of the granular material being used. As seen in Fig. 34, the horizontal section is fitted with a transverse sweeping N_2 port that causes material to flow over the lip at the opposite end of the horizontal section and to drop via gravity from that point on. Cutting off the N_2 supply lets the material assume its angle of repose in the horizontal section, which is long enough to contain the slope of material.

The 10-cm secondary burner is fitted with a 1-in.-diameter gravity-pneumatic feeder (see Fig. 34). This feeder has been used for introducing crushed TRISO fertile particles to the burner during 37 separate runs (10 to 20 kg per run). The feed rate with this type of material is ~ 10 kg/min with a N_2 flow of 23 liters/min (the minimum flow required for a nonerratic flow of material).

This feeder has been completely trouble free with no blockages or "rat-holes." No heel is left in the feeder after emptying. The feeder is capable of intermittent operation, which allows introduction of small amounts of material from a full hopper, if required. Stopping the N_2 supply results in immediate cessation of material flow.

The short time required for batching a load into the burner makes this a very attractive feed method for secondary burners. The 20-cm secondary burner will have a batch size close to 60 kg, which could be introduced in 6 min using the 1-in.-diameter feeder. This is a short time compared with the overall cycle time (5% of a 2-hr cycle).

Further work on this device for secondary burners includes trying feeders with diameters of 1-1/4 in. and 1-1/2 in. as well as trying smooth

Fig. 34. Gravity-pneumatic feeder, 10-cm secondary burner

"flow-line" butt weld fittings to reduce material holdup in the threads of the presently used screw fittings.

The secondary burner was used for some batch primary tests with the gravity-pneumatic feeder employed for charge introduction. The N₂ flow was kept at 30 liters/min with a resultant material feed rate of 6.6 kg/min. The material used was -3/16-in. graphite with 20% particles mixed in. Consistent flow was observed with no blockage problems and excellent intermittent operability.

The 40-cm primary burner could be top fed with a 1-in. feeder activated ~12% of the time to give a semicontinuous feed of ~800 g/min. Use on the 20-cm primary burner would require ~3% on-time for semicontinuous feed of 200 g/min.

4.2.1.5. Induction Heating Experiences

An induction heating system has been installed for heating the 10-cm secondary burner. To date, two different coil designs have been used to actually make burner runs. The motor-generator is a 30 kW, 10 kHz set with 440V maximum output.

The first coil used was a 53-turn, 30-in.-long solenoid coil made of 3/8-in. copper tubing. The air gap between the 4-in. Schedule 40 Hastelloy-X tube and the coil was 0.30 in. The coil was insulated on the outside only, so that the copper tubing was bare on the side toward the burner tube. The outside insulation consisted of 2 in. of Fibrefax with a stainless steel outer jacket (10-in. diameter). The end plates of the stainless jacket were 2 in. from the end coil turn. No appreciable heating of these plates was noted; apparently no heating is caused by induction, only by convection and radiation from the burner tube. The coil was

balanced by setting the autotransformer at a 0.8/1 turndown ratio and setting the capacitor at 154 kVA. The coil cooling water flow was 2.3 gal/min.

The initial heatup rate using this coil indicated that 18 kW was being dissipated in the burner tube as heat. The remaining 12 kW was taken up by resistive losses in the coil, autotransformer, capacitor, and lead line from the motor-generator set. Since these readings were taken essentially at ambient temperatures, heat losses to the surroundings were negligible. The tube could be heated to 900°C, at which time the heat losses to the coil via radiation were up to 18 kW, thus bringing the tube temperature to a steady state.

The coil was used in burner Runs 37 and 38. After combustion was stabilized with the tube at 900°C, the 18 kW of power loss to the coil with the motor-generator set off was enough to remove sufficient heat, even at peak burn rates, to keep the tube at 900°C. Because of this ample cooling capacity, no cooling air was required in the vicinity of the coil.

The second coil was built to enable 1/8 in. of asbestos insulation to be placed between the coil and the burner tube to reduce radiant losses. The coil was made with a 6.5-in. i.d. such that it could be slipped onto the burner tube over the existing flanged ends. It is 28 in. long with 12 turns (the number of turns was determined by shunting to obtain the correct impedance) and is made of 3/8-in. copper tubing. There is a 1-in. air gap between the coil and the burner tube. The coil cooling water flow is 2.3 gal/min. There is Fibrefax insulation (3 in. thick) on the outside of the coil and a 13-in.-diameter stainless steel jacket holds the insulation in place. The jacket initially had a 10-in. diameter but was susceptible too much power from the coil; therefore, it was enlarged to reduce the induced power. The jacket end plates were originally stainless steel (located 2 in. from the coil end turns). These plates became very hot due to induced

current from the coil and were therefore changed to transite, which performs well with no heating due to induction effects. The end plates are not required to be mechanically strong, but only to seal the cooling air flow; therefore, transite is acceptable.

This coil was balanced at a 0.8/1 transformer turndown and 539 kVA of capacitance. The initial heating rate with this coil indicates 12 kW being dissipated in the tube, with the remainder as coil and peripheral electrical equipment resistive losses. The steady-state tube temperature with this coil configuration is $\sim 850^{\circ}\text{C}$.

To check induced power in the support rods for the prototype burners, a test was made by embedding a thermocouple in a 1/2-in. stainless steel rod positioned 1/2 in. from the present coil. No appreciable induction heating of the rod was noted; only convective heatup was observed.

It is presently planned to have a metal susceptor surrounding the burner tube for indirect heating in the prototype burners. Tests on a similar system are planned for the 10-cm secondary burner. Materials for these tests have been ordered.

4.2.1.6. Metallurgy of Sintered Metal Filters

Sintered metal filters are currently being used in the pilot-scale fluid bed burners. The filters are cylindrical (3-in. diameter, 39 in. long, and 1/8 in. thick) and are made of type 316L sintered stainless steel with a mean pore size of 5 μm .

To determine the expected useful life of the filters, a set of filters was subjected to 30 typical burner cycles. A sample was cut from each end of the filters, representing the extremes in temperature to which the filters were subjected. During each cycle (1-1/2 hr duration), one sample was at 800°C , while the other sample was at 300°C .

The gas atmosphere was 20% CO, 70% CO₂, and 10% N₂ at 15 psia. The normal gas flow through the filters was 0.85 ft³/min/ft².

The filters were used to remove fine carbon and ThO₂ (≈40 μm) from the gas stream. To prevent carbon buildup on the filter surfaces, a backflow of gas was effected every 2 min for a short duration (1 to 2 sec).

Both samples were submitted to Metallography. The hardness of the sample exposed at 800°C was measured at DPH 250, while the 300°C sample measured DPH 140.

The following observations were made from photomicrographs:

1. The section exposed at 300°C revealed no signs of oxidation and carburization, and no signs of significant microstructural changes.
2. The section exposed at 800°C revealed significant oxidation attack (≈0.5 mil on each side). Considering that the porous structure is held together by a metal network on the order of 1 to 2 mils thick, approximately half of the "useful" life of the filter is used up. The only significant microstructural change observed in the photomicrographs was precipitation of M₂₃C₆ carbides, predominantly at grain boundaries (sensitization).

The above observations are considered normal for this material. It is not believed that the sensitization phenomenon played any role in the high rate of oxidation at 800°C.

In view of these results, the use of materials such as Hastelloy-X, which is more resistant to oxidation and carburization (in that order), is recommended. Two Hastelloy-X filters have been ordered with delivery scheduled for December 1974.

4.2.2. 20-cm Prototype Secondary Burner

The conceptual design of the 20-cm prototype secondary burner is near completion. The design is based on:

1. Process Design Criteria: 20-cm Secondary Burner (Ref. 12).
2. Analyses on (a) feed and product characterization from the 10-cm secondary burner and (b) fluidization characteristics.
3. Performance analysis of in-vessel filters in the secondary burner (Ref. 13).

The overall system layout is given in Section 7, "Systems Design," Fig. 51. Detailed design is progressing on schedule and is described later in this section.

4.2.2.1. Feed and Product Characterization

The secondary burner feed consists of crushed FSV-type (P-2 type) TRISO fertile particles burned back to the SiC coating and external carbon carried over from the primary burner. The compositions of the P-2 type TRISO fertile particles, an average secondary burner feed, and the product are given in Tables 25, 26, and 27, respectively.

Typical size distributions of the feed and product are given in Figs. 35 and 36. (A detailed analysis is given in Ref. 13.) Based on this size distribution and the information in Tables 25 through 27, the feed and product properties were estimated (Table 28).

4.2.2.2. Fluidization Characteristics

Two important parameters in fluidization, i.e., the minimum fluidization velocity U_{mf} and the terminal velocity U_t , were calculated

TABLE 25
COMPOSITION OF P-2 TYPE TRISO FERTILE PARTICLE
(BASIS: ONE FUEL ELEMENT)

Component	Weight (kg)	Wt %	Specific Gravity	Miscellaneous
Kernel	6.61	60.7	>8.4	Carbon: 9.5~12.0%
Inner PyC	0.61	5.6	1.75~1.95	1.85 ^(a)
Buffer	0.74	6.8	0.9~1.35	1.1 ^(a)
SiC	1.17	10.7	>3.18	
Outer PyC	<u>1.77</u>	<u>16.2</u>	1.70~1.90	1.8 ^(a)
	10.90	100.0		

^(a) Mean values quoted by the Fuel Group.

TABLE 26
COMPOSITION OF SECONDARY BURNER FEED^(a)
(BASIS: ONE FUEL ELEMENT)

Component	Weight (kg)	Wt %	Specific Gravity
Fertile particles ^(b)	9.13	89.6	4.23
Kernel			
ThC ₂	6.51		8.4
Free carbon	0.10		
Inner PyC	0.61		1.85
Buffer	0.74		1.1
SiC	<u>1.17</u>		3.18
	9.13		
External carbon ^(c)	<u>1.06</u>	<u>10.4</u>	2.0
	10.19	100.0	3.79 avg.

(a) From Ref. 14.

(b) Particles are burned back to SiC coat in the primary burner.

(c) Primary burner product contains 5% burnable carbon external to the particles. (The goal is to reduce it to 2% level.)

TABLE 27
COMPOSITION OF SECONDARY BURNER PRODUCT^(a)
(BASIS: ONE FUEL ELEMENT)

Component	Weight (kg)	Wt %	Apparent Density
ThO ₂	6.71	83.46	9.104
SiC	1.17	14.54	3.180
2% burnable carbon ^(b)	<u>0.16</u>	<u>2.00</u>	<u>2.000</u>
	8.04	100.00	6.790 avg

^(a) From Ref. 14.

^(b) Defined by the Design Criteria (Ref. 12). A goal is set to reduce this to the 0.5% level (Ref. 15).

Fig. 35. Feed size distribution, 20-cm prototype secondary burner

Fig. 36. Product size distribution, 20-cm prototype secondary burner

TABLE 28
PROPERTIES OF FEED AND PRODUCT

	Feed	Product
Average size, μm	228	56
Theoretical average density, g/cm^3	4.62	7.18
Porosity, %	17.9	5.5
Apparent density, g/cm^3	3.79	6.78
Voidage (ϵ_o)	0.55	0.65
Bulk density, g/cm^3	1.70	2.40
Sphericity	0.677	0.672

using the modified Carman-Kozeny equation (Refs. 16,17) and Stoke's equation (Ref. 16):

$$U_{mf} = \frac{\epsilon_{mf}^3}{5(1 - \epsilon_{mf})} \frac{(\rho_s - \rho_f)g}{S^2 \mu_f} \quad \text{for } Re_p < 20 \quad , \quad (1)$$

$$U_t = \frac{0.153 d_p^{1.14} g_c^{0.71} (\rho_s - \rho_f)^{0.71}}{\mu_f^{0.43} \rho_f^{0.29}} \eta \quad \text{for } 2.0 < Re_p < 500 \quad (2)$$

where

$$S = \frac{6}{\phi_s d_p} \quad ,$$

$$\phi_s \text{ (sphericity)} = \frac{\text{surface area of sphere (same volume)}}{\text{surface area of particle}}$$

$$\eta = 0.843 \log \frac{\phi_s}{0.065} \quad ,$$

d_p = particle diameter,

ρ_s = particle density,

ρ_f = fluidizing gas density ($3.32 \times 10^{-4} \text{ g/cm}^3$),

μ_f = fluidizing gas viscosity ($5.2 \times 10^{-4} \text{ g-cm/sec}$),

$\epsilon_{mf} = 1.1\epsilon_o = 0.59 \text{ (feed),}$
 $\phantom{\epsilon_{mf}} = 0.68 \text{ (product),}$

$$Re_p = \frac{d_p \rho_f u}{\mu_f} \quad ,$$

u = superficial velocity.

The values calculated using the properties of the feed and product in Table 28 are given in Table 29.

The quality of fluidization is often measured by the following criteria (Refs. 18,19):

$$Fr_{mf} = \frac{U_{mf}^2}{d_p g} \begin{cases} Fr_{mf} < 0.13, \text{ particulate fluidization} \\ Fr_{mf} > 1.3, \text{ bubbling fluidization} \end{cases}, \quad (3)$$

$$\left(Fr_{mf} \right) \left(Re_{p,mf} \right) \left(\frac{\rho_s - \rho_f}{\rho_f} \right) \left(\frac{L_{mf}}{d_t} \right) \begin{cases} < 100 \text{ particulate} \\ > 100 \text{ bubbling} \end{cases}, \quad (4)$$

where L_{mf} = bed height at minimum fluidization,

d_t = tube diameter.

Table 30 is a comparison of these values for the feed and product. Table 30 shows that the fluidization is in the bubbling region at the beginning and approaches a particulate region at the end. However, these results should be interpreted with caution, since the fluidization of particles of mixed sizes is very difficult to predict. In fact, the experimental results in the 10-cm secondary burner show a heavy slugging motion in the bed. Stewart's criteria (Ref. 20) predict a slugging fluidization for this burner due to the high gas velocities and small particle size. A detailed discussion on burner slugging is given in Refs. 21 and 22. The results only are summarized for three different superficial velocities in Table 31.

From a comparison of theory and data, it appears that the secondary burner operates in the slugging region. No operational problems have been observed from operating in this region since the vessel height has been designed to accommodate the bed.

TABLE 29
 U_{mf} and U_t FOR FEED AND PRODUCT

	d_p (μm)	ρ_s (g/cm ³)	U_{mf} (cm/sec)	U_t (cm/sec)
Feed				
Average	228	3.79	4.8	177
ThC ₂	333	8.4	22.7	837
SiC	140	3.18	1.52	56
C	127	1.57 ^(a)	0.62	23
Product				
Average	56	6.79	0.97	26
ThO ₂	52	9.1 ^(a)	1.13	22
SiC	85	3.18	1.05	21

(a) Estimated apparent densities.

TABLE 30
QUALITY OF FLUIDIZATION

	U_{mf} (cm/sec)	Fr_{mf}	$(Fr_{mf}) (Re_{p,mf}) \left(\frac{\rho_s - \rho_f}{\rho_f} \right) \left(\frac{L_{mf}}{dt} \right)$
Feed			
Average	4.8	1.03	>>100
ThC ₂	22.7	15.8	>>100
SiC	1.52	0.17	~120
C	0.62	0.03	<100
Product			
Average	0.97	0.17	74
ThO ₂	1.13	0.25	157
SiC	1.05	0.27	90

TABLE 31
SLUGGING FLUIDIZATION

	Feed			Product		
Equivalent tube diameter, cm	18.2			18.2		
Minimum fluidization velocity, cm/sec	4.8			1.0		
Bulk density of bed material, g/cm ³	1.7			2.4		
Bed weight, kg	60.0			47.3		
Bed height at minimum fluidization, cm	120.0			67.0		
Bed ΔP , cm H ₂ O	186.0			147.0		
Relative slug velocity, cm/sec	66.1			66.1		
	u (a) = 30	u = 60	u = 90	u = 30	u = 60	u = 90
Absolute slug velocity, cm/sec	91.3	121.3	151.3	95.1	125.1	155.1
Relative slug volume (b)	0.38	0.84	1.29	0.44	0.89	1.35
Emulsion length factor (T)	2	4	4	1	2	2
Emulsion length (TD), cm	36.4	72.8	72.8	18.2	36.4	36.4
Slug length, cm	27.0	99.3	152.5	20.8	63.1	95.8
Maximum bed height, cm	166	221	275	97	127	158
Minimum bed height, cm	152	160	181	89	95	109
Fluctuation of bed height, cm	14	61	94	8	32	49
Pressure fluctuation due to slugging, cm H ₂ O	26	77	97	27	69	85
Time between slug eruptions, sec	0.7	1.4	1.5	0.4	0.8	0.85

(a) u = superficial velocity, cm/sec.

(b) $K = \frac{u - U_{mf}}{u_B}$; Stewart criterion: $K > 0.2$ for slugging fluidization.

4.2.2.3. Design Conditions

The design conditions for the burner main vessel and the burner components are given in Table 32. All burner components will be designed to meet the ACC System Design Description (SDD) component design pressures and temperatures. The burner vessel will be designed for a standard hydrostatic test in accordance with the ASME Boiler and Pressure Vessel Code, Section VIII, Pressure Vessels, and will be designed to prevent overpressurization.

4.2.2.4. Burner Vessel

The burner vessel is divided into five sections, i.e., the gas plenum, the burning section, the disengaging section, the in-vessel filter chamber, and the plenum to accommodate the filter blow-back nozzles and off-gas exit. The disengaging section and the filter chamber are constructed as one piece; the other sections can be separated by the semi-remotely operated flange assemblies, shown as clamps 1, 2, and 3 in Fig. 37.

The bottom section of the burner, which is tapered 10° from a vertical line, has the following advantages:

1. Eliminates the stagnant zone at the corners above the distributor plate (Ref. 23) that causes hot spots.
2. Induces a circulatory motion (Ref. 24).
3. Increases superficial velocity at the bottom. This further eliminates stagnation and enables use of more holes in the distributor plate (more uniform distribution) for the same pressure drop.
4. Enhances axial heat transfer from the reaction zone at the bottom.

TABLE 32
DESIGN CONDITIONS

Burner main vessel

Average burn rate	Minimum 1 FE/hr (50 g C/min)
Design temperature	900°C
Design pressure	Maximum 24 psia
Startup	By induction heating
Cooling	Wall cooling by forced air
Operational mode	Batch
Charge/cycle	60 kg
Solid transport	Pneumatic
Fluidizing gas	Oxygen
Diluent gas	N ₂ or CO ₂

Burner components

Feed hopper, maximum 24 psia	<450°C
Cooling shroud, -10 in. WG to 5 psig	900°C
Cooling air filter, 10 in.H ₂ O $\Delta P_{\max}$	900°C
Off-gas filter	900°C

Fig. 37. 20-cm secondary burner

5. Enables removal of the induction coil and susceptor plate assembly from the burner without increasing the gap for cooling air, which deteriorates the cooling capacity.

Other burners with this tapered bed design have been successfully used elsewhere (Refs. 25,26).

4.2.2.5. In-Vessel Filters

The performance of in-vessel filters in secondary burners is analyzed in Ref. 13. Based on this analysis, in-vessel filters are employed in the 20-cm secondary burner.

There are six Hastelloy-X sintered metal filters, divided into three groups with each group having two filters. Each group is separated from the other groups by plates. The three groups of filters will be back-blown alternatively, as shown in Fig. 38. The physical dimensions of the filter are given in Table 33.

TABLE 33
SINTERED METAL FILTERS

Material	Hastelloy-X
Total surface area	8.9 ft ²
Diameter	2-3/4 in.
Length	18 in.
Thickness	1/4 in.
Mean pore size	5 μm (Grade H)
Nominal removal ratings (98%).	0.4 μm
Absolute removal ratings (100%).	1.0 μm

Blow-back of the filters will be accomplished by using the recycled off-gas (compressed to 60 to 80 psi) through a venturi nozzle. Detailed drawings will be provided in a later report.

6	9	METAL FILTER 2 1/4 DIA x 18 LONG	SEE NOTE 1.
1	8	THRU CENTER DUCT	
6	7	VENTURI	
6	6	JET TUBE	
1	5	THERMOCOUPLE TUBE	
1	4	TOP CAP PLATE 12.75 DIA x .50	
1	3	MOUNTING PLATE 11.75 DIA x .375	
3	2	SEPARATOR PLATE 21.25 x 8.0 x .187	
1	1	TUBE & FILTER WELDMENT	
LIST OF MATERIAL			
ITEM	PART NO	DESCRIPTION	MATL

Fig. 38. Tube and filter assembly

4.2.2.6. Distributor Plate

A flat distributor plate is preferred for the secondary burner for the following reasons:

1. A flat distributor plate has been successfully used in the 10-cm secondary burner.
2. A stagnation zone above the distributor plate can be minimized using a tapered bottom, which adds other advantages to the system as discussed earlier.
3. Secondary burner bed materials are very fine powders and the channeling tendency is higher for the cone distributor.
4. Accommodation of the bed discharge mechanism, which is discussed below, is easier with a flat plate.

The effect of a distributor plate diminishes as the bed height increases. In a 20-cm burner, bed slugging is inevitable regardless of the distributor design. The design is, therefore, aimed to provide the most uniform distribution of gas at the bottom of the bed and to reduce the possibility of stagnation. The holes are distributed evenly with a triangular pitch, which provides the most even solid circulation pattern.

A product discharge valve is incorporated in the distributor plate. Since the area occupied by the valve is 3% of the total area, stagnation of solids at this spot is very unlikely. Detailed dimensions and the hole arrangement are shown in Fig. 39.

DIAMETER: 6-1/8 in. (155.6 mm)
 $d_{or} = 1.4$ mm (55 mils)
 120 HOLES
 $\Delta P = 35$ cm H_2O
 TRIANGULAR PITCH: 14 mm
 OPEN AREA = 0.96%

Fig. 39. Distributor plate, 20-cm prototype secondary burner

4.2.2.7. Pneumatic Feeder

A gravity-pneumatic feeder has been successfully used in the 10-cm secondary burner. Test results for this feeder shows that a 1-in. feeder will be adequate for the 20-cm secondary burner (Ref. 27). The estimated flow rate is 10 kg/min, and it will take 6 min to introduce a batch size of 60 kg. A minimum of 23 liters/min of N_2 is required for a nonerratic flow of material (Ref. 27). The gravity-pneumatic feeder is the same as that shown in Section 4.2 (Fig. 34) for the 10-cm secondary burner.

4.2.2.8. High-Temperature Product Removal Valve

The basic arrangement of the product removal valve is similar to that used in the 10-cm secondary burner (Ref. 28), except that the valve is located on the distributor plate instead of on the side wall. Location of the discharge valve on the side wall is unpractical for the following reasons:

1. The use of remote flanges makes it necessary to locate the valve high above the distributor plate, which makes it difficult to withdraw stagnant materials on the distributor plate.
2. Due to the larger burner size, it is difficult to withdraw bed materials at the far corners from the valve.
3. With the valve on the side wall, it is more difficult to design an induction heating coil assembly that can be remotely removed from the burner tube.

Location of the product removal valve on the distributor plate alleviates the above difficulties and also reduces the effect of jet penetration, since the materials flow from top to bottom. The burner bottom section, including the high-temperature bed removal valve, is shown in Fig. 40.

Fig. 40. 20-cm secondary burner base

4.2.2.9. Induction Heating for Startup

During normal operation, the bed charge will be dumped into the burner tube while it is still hot. For a worst case, however, it is assumed that heatup of the whole bed from ambient temperature to 900°C can be accomplished in 1 hr. A preliminary estimation shows that a 36-in.-long induction heating section with 100-kW capacity will be sufficient for this purpose.

Detailed transient heatup response and induction coil design studies are under way. Intermediate results shows that radiation loss into the induction coil will be substantial without insulation of the coil. The insulation material and the induction coil design will be determined in a direction to minimize the conduction loss and electrical loss to the coil. The use of a 1/8-in. susceptor plate shows substantial advantages over the case without a susceptor plate in terms of both the coil electrical efficiency and the conduction loss.

4.2.2.10. Burner Cooling

Burner cooling will be accomplished by forced air through cooling shrouds around the tube. A preliminary calculation indicates that the surface area provides sufficient cooling capacity for the heat generated in the burner.

4.2.2.11. Remote Maintenance

The burner assembly and the semi-remote flanges are shown in Fig. 41 and are discussed in Section 7, "Systems Design." It is worthwhile, however, to mention that the flange design employs thermal shrouds to protect the seal rings from thermal shock. As a result of this design change, the distributor plate in Fig. 39 has been detached from the seal ring and is mounted directly on the lower flange. The thermal shroud and the distributor plate are shown in Fig. 41.

NOTES:

- △ ITEM TO BE SUPPLIED BY GRAY TOOL CO.
 2 DENOTES .375 REF. FLANGE THICKNESS
 ON ITEMS 6, 7, & 8 SEAL RINGS.

-1 VESSEL ASSEMBLY

REQD/ASSEMBLY	ITEM	PART NO.	DESCRIPTION	MATL
	11	48506	CLAMP	
	10	49538	CLAMP	
	9	70438	CLAMP	
	8	51279	SEAL RING	
	7	66237	SEAL RING	
	6	66233	SEAL RING	
	5	2450007-1	LOWER CAP	
	4	2450003-1	LOWER VESSEL	
	3	2450003-2	UPPER VESSEL	
	2	2450003-3	UPPER CAP	
	1	2450007-1	VESSEL ASSEMBLY	
REQD/ASSEMBLY	ITEM	PART NO.	DESCRIPTION	MATL

Fig. 41. Vessel assembly showing semi-remote flanges

5. LEACHING

5.1. INSPECTION OF CORROSION COUPONS INSTALLED IN THE 13-CM-DIAMETER LEACHER

Early this year a program designed to indicate "gross" effects of corrosion within the dissolution system was begun. Corrosion coupons of four candidate metal alloys (Incoloy 690, Incoloy 825, 304L stainless steel, and Armco 22-13-5 stainless steel) were installed on January 29, 1974 in various locations within the 13-cm-diameter leacher.

The methods by which the coupons were fabricated, installed, and initially inspected are discussed in this section. Coupon exposure history, calculated corrosion rates, and general comments made from a visual inspection are also presented. Some initial information (reported previously in Ref. 1) is included for completeness.

5.1.1. Summary of Results

Initial inspection indicates no major surprise with regard to adverse corrosion rates in a close-to-actual operating environment. The corrosion rate measured for the 304L stainless steel coupons was 8 to 18 mils/yr, which is comparable to rates reported in the literature (Refs. 29-31).

Perry (Ref. 32) suggests that the first 48 hr of exposure be disregarded in corrosion rate determinations. This initial period allows for any film formation on the metal and therefore eliminates inaccuracies generated for comparative work. It is for this reason that firm conclusions should not be drawn on this initial data.

Further routine inspections will be conducted at timely intervals and are expected to generate more conclusive data. The next inspection will be conducted after 50 hr of exposure and will be a base for comparison with future data. Results of future inspections will be reported as they are obtained.

5.1.2. Discussion

5.1.2.1. Corrosion Study Basis

The corrosion studies initiated for the HTGR fuel recycle dissolution system are intended to provide a means of determining gross effects on various metal alloys at near-actual operating conditions. These studies are primarily to discern whether any adverse effects can be expected due to our particular leacher operating environment.

Discussions and guidance from Allied Chemical representatives at ICPP resulted in mutual agreement that detailed corrosion studies need not be undertaken. This conclusion was reached based on the relatively large amount of existing corrosion data contained in the literature on a variety of metal alloys in a Thorex environment. Table 34 presents some of the existing corrosion data on various metal alloys in nitric acid/Thorex solutions.

With this general guideline, along with guidance from ACC corrosion engineers, the corrosion coupons were fabricated and installed in the 13-cm-diameter leacher.

5.1.2.2. Coupon Preparation

Corrosion coupons from samples of Incoloy 825, Incoloy 690, 304L stainless steel, and Armco 22-13-5 stainless steel were prepared for

TABLE 34
SELECTED CORROSION DATA FROM LITERATURE
ON METAL ALLOYS IN NITRIC ACID/THOREX SOLUTIONS

Process/Environment Description	Metal Alloy	Specimen Location	Exposure Time (hr)	Corrosion Rate (mils/yr)
ORNL Thorex plant, batch dissolver, approximately 13M HNO_3 , 0.04 to 0.33M Hg (Ref. 30)	304L SS	Liquid	2706	31.8 ± 0.9
	304L SS	Vapor	2706	40.3 ± 1.5
	309S Cb	Liquid	2706	15.0 ± 1.6
	309S Cb	Vapor	2706	34.9 ± 1.9
Battelle scouting evaluations in Saltex Thorex process, boiling initial Thorex, 13.0M HNO_3 , 0.05M F^{-1} , 0.20M Al^{+3} (Ref. 31)	Inconel 700	Liquid	24	252
		Vapor	24	528
	Incoloy 804	Liquid	24	15.6
		Vapor	24	14.4
	319L SS	Liquid	24	24.0
		Vapor	24	27.6

installation in four different locations within the leacher. The coupons were fabricated using ASTM designation G 4-48 as a general guideline. Table 35 provides information on the metal alloys.

Each coupon was cut lengthwise down the center and then rejoined using a TIG 300/300 Lincoln arc welder set for direct current, straight polarity, and an argon purge of 12 SCFH. Both Incolloys were welded using a 1/16-in.-diameter 2% thorium electrode; the stainless steels were welded with a 3/32-in.-diameter 2% thorium electrode. The Incoloy coupons were welded using a 1/16-in. gap butt weld joint, and the stainless steel coupons were welded with a 1/16-in. gap 80° V-groove joint.

Welding of the coupons was accomplished using a gas lens, high-frequency start, 1/4-in. extended electrode with the torch held at 90° to the surface. Agitation of the puddle was avoided and the weld root was left unshielded. All coupons were then milled and polished with 120 grit on the front and back faces.

5.1.2.3. Coupon Location

Four sets of coupons are installed within the 13-cm-diameter leacher to obtain maximum information. The first set is located at the bottom of the leacher vessel where it is exposed to maximum temperature, agitation, solids contact, and liquid aeration. The second set is located approximately at the midpoint of the leaching vessel where it comes in contact with the liquid-vapor interface. Location of the interface is, however, dependent on the particular experiment being conducted. The third set of coupons is located at the top of the leacher vessel and encounters primarily the vapor phase and any entrained liquid. The fourth and final set is located at the entrance of the down-draft condenser, also in contact with the vapor phase. Location and labeling of the coupons are shown in Fig. 42.

TABLE 35
CORROSION COUPON ALLOYS USED IN
13-CM-DIAMETER LEACHER

Alloy	Heat No.	Density (g/cm ³)	Stock Type	Weld Metal Filler
Incoloy 825	HH4229F	8.14	Sheet	Incoloy 65
Incoloy 690	NX10CLH	8.14	Sheet	Incoloy 625
304L SS	20316	8.02	Sheet	308 ELC
Armco 22-13-5 SS	031028	7.88	Bar	Armco 22-13-5

Fig. 42. 13-cm-diameter leacher showing position of related equipment and corrosion coupons

Support and separation of the coupon sets were accomplished by overlaying 3/8-in. and 7/16-in. tygon tubing on a piece of 1/4-in.-i.d. stainless steel tubing. These assemblies were in turn supported by Gyrolok fittings between the nitrogen purge legs in the vessel and a support structure in the condenser. The support structures are shown in the photographs in Figs. 43 through 45.

5.1.2.4. Corrosive Conditions

The corrosive conditions to which the metal alloys are exposed consist of a Thorex - burner ash mixture at its atmospheric boiling point ($\sim 118^{\circ}\text{C}$). The Thorex is a mixture of 13M HNO_3 , 0.1M $\text{Al}(\text{NO}_3)_3$, and 0.05M HF . Solids charged to the leacher are predominantly secondary burner ash resulting from the combustion of crushed TRISO coated thorium carbide particles, i.e., thorium oxide, residual carbon, and silicon carbide hulls. The burner ash - Thorex mixture is air sparged to promote solids suspension and thus enhance dissolution rates. In some experimental tests, the burner ash may also contain whole fissile particles, either actual or simulated. Typical size distributions of solids utilized in experimental runs are presented in Table 36.

5.1.2.5. Inspection After Initial Exposure

Prior to their inspection, the corrosion coupons were exposed to the corrosive conditions for approximately 22 hr. A more detailed history of the coupon exposure is given in Table 37.

Visual examination of the coupons was conducted after cleaning with a mild soap solution, drying, and weighing the specimen. Inspection was performed with the aid of a microscope at 3X magnification. Results of the inspection and further comments are given in Table 38.

(a)

(b)

Fig. 43. F-series coupons and mounting bracket: (a) coupons on bracket, and (b) coupons and bracket placed in condenser of 13-cm-diameter leacher

(a)

(b)

Fig. 44. Mounting bracket for coupons: (a) coupons in positions 2 and 3, and (b) coupons in position 1

(a)

(b)

Fig. 45. Positions of coupon brackets as mounted on dip legs of 13-cm-diameter leacher

TABLE 36
TYPICAL SOLID SIZE DISTRIBUTIONS
OF SECONDARY BURNER ASH

Weight Percent Less Than	Secondary Burner Ash	SiC Hulls in Burner Ash	Simulated Fissile Glass Beads
10 microns	12	2	-
25 microns	39	5	-
50 microns	53	10	-
75 microns	65	20	-
100 microns	75	45	-
150 microns	90	65	-
200 microns	97	93	-
250 microns	99	98	5
300 microns	100	100	40
400 microns	-	-	100

TABLE 37
CORROSION COUPON EXPOSURE HISTORY
IN 13-CM-DIAMETER LEACHER^(a)

Leaching Run	Feed Identification	Boiling Point (°C)	Pressure (psia)	Total Time in Leacher (hr)	Time at B.P. (hr)
79	TRISO fertile ash plus glass beads in Thorex	118	14.7	3.7	2.5
80	TRISO fertile ash plus glass beads in Thorex	118	14.7	5.5	3.5
81	TRISO fertile ash plus glass beads in Thorex	118	14.7	8.5	6.5
82	TRISO fertile ash in Thorex	118	14.7	3.0	1.0
83	TRISO fertile ash in Thorex	118	14.7	3.5	2.0
84	TRISO fertile ash plus glass beads in Thorex	118	14.7	<u>8.0</u>	<u>6.5</u>
				32.2	22.0

(a) Start of run Thorex solution consists of 13.0M HNO₃, 0.05M HF, and 0.1M Al(NO₃)₃ - end of run Thorex solution consists of approximately 9.0M HNO₃, 0.05M HF, 0.1M Al(NO₃)₃, and 1.0M Th(NO₃)₄. Minimum temperature (ambient) ≈ 25°C; maximum temperature (B.P.) ≈ 120°C.

TABLE 38
VISUAL INSPECTION OF CORROSION COUPONS AFTER 22 HR OF EXPOSURE

INCOLOY 825

1. All samples had some rough areas, which were due to inadequate milling.
2. There was dark discoloration around the support holes of the B and F coupons.
3. Some SiC hulls were wedged in crevices left by the inadequate milling in the A coupon.
4. In general, the coupons were only slightly discolored and no apparent effects of pronounced corrosion were evident.

INCOLOY 690

1. There were rough areas on the coupons due to inadequate milling. These appeared over the wrought metal surface, as well as the weld bead interface where the metals has not been milled flush to one another.
2. In general, the wrought metal was slightly tarnished and the weld metal fairly bright, making the weld outline quite clear. There was also more pronounced discoloration around the support holes.

304L STAINLESS STEEL

1. All coupons had a brownish discoloration from heat, the wrought metal being more discolored than the weld metal. The F coupon was not as discolored as the others.
2. Again, inadequate milling of the coupons left rough areas in some places. The B coupon had three small holes in the weld bead, which were existent prior to exposure.

ARMCO-22 STAINLESS STEEL

1. In general, all coupons seemed bright and in good condition.
2. On the A and B coupons, a small knife line at the wrought-weld interface was apparent. This may have been caused by inadequate milling but cannot be established from the photographs. This item will be checked on the next inspection.

Following the inspection, each coupon was photographed from both faces. Figure 46 shows photographs of a typical coupon before and after the 22-hr exposure.

Data pertaining to the weight loss and the resultant corrosion rate of each coupon are tabulated in Table 39.

In general, a slight heat discoloration, as well as a small weight loss, was experienced in all of the corrosion coupons.

During the visual inspection, it was found that some of the coupons had not been milled to produce a completely flush surface. This resulted in some of the small silicon carbide shells adhering to and wedging in small crevices. The reported weight differences in Table 39 are therefore subject to some scrutiny.

There were no apparent indications of knife-line attack at the metal-weld bead interface, weld bead decay, and grain attack, nor of crevice attack at the support structure interface. Although the edges of the coupons were left unmilled and unpolished, there was no apparent indication of corrosion at those locations.

After inspection, the coupons were reinstalled in the 13-cm-diameter leacher in approximately the same locations. Tygon tubing was replaced with teflon spacers due to the embrittlement of the tygon in the acidic atmosphere. Another inspection will be conducted after at least 50 hr of exposure has been experienced by the coupons.

Due to the short exposure period, along with the very small weight losses occurring in the coupons, it does not appear feasible to draw any firm correlations and/or conclusions from the data obtained in this initial inspection. Future inspections of these same coupons will allow a firmer basis upon which such correlations and conclusions can be achieved.

(a)

(b)

Fig. 46. Typical coupons (304L SS) before and after 22 hr of exposure to Thorex - burner ash solution: (a) before exposure, and (b) after exposure

TABLE 39
CORROSION RATES OF COUPONS INSTALLED IN
13-CM-LEACHER AFTER AN EXPOSURE OF 22 HR

Coupon Identification	Location	Surface Area (in. ²)	Weight Change (g)	Corrosion Rate (mils/yr)
Incoloy 825				
F	Condenser	7.573	0.022	8.4
A	Vapor	7.629	0.046	17.4
B	Interface	7.707	0.056	21.0
C	Liquid	7.525	0.062	23.8
Incoloy 690				
F	Condenser	7.319	0.099	39.1
A	Vapor	7.252	0.040	15.9
B	Interface	7.299	0.031	12.3
C	Liquid	7.335	0.043	17.0
Stainless 304L				
F	Condenser	8.082	0.020	7.3
A	Vapor	8.136	0.045	16.3
B	Interface	8.027	0.051	18.7
C	Liquid	8.071	0.067	24.4
Stainless Armco 22-13-5				
F	Condenser	13.503	0.080	18.0
A	Vapor	13.525	0.108	24.2
B	Interface	13.029	0.096	22.3
C	Liquid	13.814	0.124	27.2

5.1.3. General Conclusions and Recommendations

In general, the corrosion rates measured on the coupons in the 13-cm-diameter leacher do not seem abnormal to cited literature values.

Future inspections should be made at regular intervals to provide reliable and conclusive data. If additional controls and/or standardization of the corrosion tests are required, they should be coordinated through the General Atomic Metallurgical Department. It is recommended that pilot plant testing continue with the current "gross indicator" program.

5.2. RUN SUMMARY FOR LEACHING RUNS 72-76, 78-81, and 84

5.2.1. Summary

Leaching Runs 72-78, 78-81, and 84 were conducted to investigate the dissolution of crushed burned-back TRISO fertile particles in the presence of whole SiC coated fissile particles. This flowsheet option could exist in a head-end reprocessing flowsheet, as described previously (Ref. 33). It was established that the presence of the whole particles adversely affects the overall batch dissolution rate and increases the difficulty of subsequent liquid-solid separation. The results are shown in Fig. 47.

5.2.2. Run Conditions

Ten test runs were conducted with the 13-cm-diameter leacher. All of the tests were made on a batch basis using a mixture of whole particles and secondary burner product (resulting from burning crushed TRISO ThC_2 particles). The size distribution of the burner ash utilized in Runs 72-76 and 78 (Ref. 34) is provided in Fig. 48. The size distribution information for the burner ash utilized in Runs 79-81 and 84 is also given in Fig. 48.

Fig. 47. Dissolution of secondary burner ash in presence of whole TRISO fissile particles

Fig. 48. Particle size distribution in leaching runs

All tests were conducted with about 3.25 liters of Thorex 13M HNO_3 /0.05M HF /0.1M $\text{Al}(\text{NO}_3)_3$ per kilogram of the secondary burner product. Operating data are shown in Table 40.

Runs 72 and 73 were made using 4/1 Th/U-238 TRISO coated particles as the whole particle portion of the feed. The size distributions and chemical analysis for these particles are included in Tables 41 and 42, respectively. Runs 74-76, 78-81, and 84 were made using glass beads to simulate the whole fissile particles. The glass beads had a size distribution of 45 wt % of 250 to 300 μm particles and 55 wt % of 450 to 500 μm particles. All tests were conducted with 1.88 kg of whole particles and 2 kg of secondary burner ash.

The liquid-solid separation of the leacher product was accomplished with a batch centrifuge. Centrifuge data for all runs are as follows:

1. 30-cm-diameter perforate basket.
2. Polypropylene filter bag (5 to 6 μm openings).
3. 1100 gravities purging force at basket sheet (2500 rpm).
4. Filter cake spun dry for about 5 min after washing.

The quantity and specific gravity of liquids in all storage tanks and the leacher were continuously and automatically monitored and recorded. Tank calibration relationships are not utilized for material balance purposes. All volumetric determinations for liquids were accomplished with graduated cylinders. Analyses of samples submitted to the Analytical Chemistry Department are given in Table 43. These data were utilized in material balance calculations (see Table 44).

TABLE 40
LEACHER OPERATING DATA, 13-CM-DIAMETER LEACHER

	72(a)	73	74	75	76	78	79	80	81	84
Whole particles charged, kg	1.88	1.88	1.88	1.88	1.88	1.88	1.88	1.88	1.88	1.88
Burner ash charged, kg	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00
Thorex charged, liters	6.50	6.50	6.50	6.50	6.50	6.50	6.50	6.50	6.50	6.50
Air sparge rate, liters/min	4.72	14.16	14.16	14.16	14.16	14.16	14.16	5.66	5.66	5.66
Leaching time at boiling point, hr	1.0	1.0	1.0	4.0	1.0	6.0	2.5	3.5	6.5	6.5
Insolubles after leach (dry wt), ^(b) g	2362.0	2361.0	2592.0	2198.0	2547.0	2362.0	2051.0	2233.0	2352.0	2139.0
Mother liquor, ^(c) liters	6.930	7.575	6.525	11.196	9.374	-	8.43	9.738	9.096	10.837

(a) Leach run number. Leach Run 77 was a cleanup run for about 10 previous runs, i.e., a run to remove any soluble Th from insoluble SiC hulls. Leach Runs 82 and 83 were for TRISO ThC₂ particles alone; a separate run summary including these runs will be issued.

(b) Includes whole particles.

(c) Includes some wash water.

TABLE 41
 SIZE DISTRIBUTION OF TRISO FISSILE PARTICLES USED
 IN LEACH RUNS 72 AND 73 (OUTER PyC REMOVED
 IN SECONDARY BURNER RUN F4RHB M-25)

<u>Screen Fraction (microns)</u>	<u>Wt %</u>
420 - 495	44.45
350 - 420	49.53
250 - 350	6.02

TABLE 42
 CHEMICAL ANALYSIS OF TRISO FISSILE PARTICLES

Compound	Wt % ^(a)	
	Sample 1	Sample 2
Th	16.05	35.72
U	4.15	9.09
SiC	22.76	24.17

(a) Based on weight as uncrushed TRISO fissile particle with outer PyC removed, i.e., as fed to leacher.

TABLE 43
SAMPLE ANALYSIS RESULTS, 13-CM-DIAMETER LEACHER

	72(a)	73	74	75	76	78	79	80	81	84
Burner ash, wt % Th	71.3	71.3	71.3	71.3	71.3	71.3	69.56	69.56	69.56	69.56
Insolubles, (b) wt % Th	12.28	11.79	14.36	0.89	11.50	1.5	4.32	2.27	5.87	2.53
Insolubles, wt % U	1.96	1.75	-	-	-	-	-	-	-	-
Mother liquor, (c,d) g Th/liter	169.3	176.20	164.0	120.8	135.3	261.9	148	136.35	134.7	126.28
Mother liquor, (c,d) g U/liter	0.08	0.14	-	-	-	-	-	-	-	-

(a) Leach run number.

(b) "Insolubles" are not crushed prior to analysis; thus, analysis indicates only metal content. Insols are leached with a Thorex-type solution and the resulting solution is analyzed for Th colorimetrically.

(c) Mother liquor analysis is based on gravimetric determination by oxalate precipitation.

(d) Includes some wash water.

TABLE 44
THORIUM MATERIAL BALANCE RESULTS, 13-CM-DIAMETER LEACHER

	72(a)	73	74	75	76	78(b)	79	80	81	84
Thorium input, g										
Burner ash	1426.0	1426.0	1426.0	1426.0	1426.0	1426.0	1391.0	1391.0	1391.0	1391.0
Thorium output, g										
Mother liquor ^(c)	1173.0	1335.0	1070.0	1352.0	1133.0	-	1248.0	1328.0	1225.0	1368.0
Insolubles	<u>290.0</u>	<u>278.0</u>	<u>372.2</u>	<u>19.56</u>	<u>293.0</u>	<u>19.13</u>	<u>89.0</u>	<u>51.0</u>	<u>138.0</u>	<u>54.0</u>
Total output	1463.0	1613.0	1442.0	1372.0	1426.0	-	1337.0	1379.0	1363.0	1422.0
Material balance closure, ^(d) wt %	102.6	113.0	101.1	96.2	100.0	-	96.1	99.1	98.0	102.2
Thorium recovery, ^(e) wt %	82.2	93.6	74.2	98.5	79.45	98.66	93.3	96.3	89.9	96.2

(a) Leach run number.

(b) Mother liquor analysis suspected of error and quantity of mother liquor was unrecorded.

(c) Includes some wash water.

(d) Output/input.

(e) Based on outlet quantities.

5.2.3. Results and Discussion

The addition of whole fissile particles reduces the overall batch dissolution rate for crushed burned-back TRISO ThC_2 particles. This is thought to be the result of ineffective agitation of leacher contents. Specifically, the results indicate that under similar conditions utilized in past tests (Ref. 34, p. 107), the presence of uncrushed particles increases the overall time required for dissolution of the exposed thorium oxide. Further testing should be conducted to determine the degree to which the dissolution rate of this type burner ash is affected by equipment and operating parameter modifications.

The TRISO coats on the fissile particles are being damaged at some point within the dissolution system (see U analysis, Table 43). It is suspected that the breakage occurred within the liquid-solids separation step, based on the following observations:

1. Two samples of the whole fissile particles were tested by Analytical Chemistry and indicated only 0.87 to 1.43 wt % of the particles were broken prior to feeding into the leacher.
2. The quantity of uranium found in the mother liquor after the leach corresponded to 0.32 to 1.36 wt % broken fissile particles.
3. The soluble uranium contained in the insols indicated that from 2.41 to 59.3 wt % of the fissile particles were broken after the liquid-solid separation step.

Further testing should be conducted to study this phenomenon.

Problems were encountered with the liquid-solid separation step in all of the leaching runs. The high concentration of solids in the initial

slurry fed to the centrifuge resulted in excessive vibration of the centrifuge. It is felt that difficulty in feeding the high-solids-content slurry at a consistent flow rate caused "off-loading" of the centrifuge. The use of a dilution tube to reduce the solids concentration (and therefore "off-loading") has been described previously (Ref. 34, p. 121). The problem will be studied further.

In Runs 73 and 79, there was no flow of slurry to the centrifuge when the dump valve was opened. It was necessary to apply heat to the bottom section of the leacher cone. This seemed to reduce the apparent plug and allowed the leacher contents to be dumped.

As a result of the centrifuge "off-loading" mentioned above, it was necessary to stop the slurry removal before all of the leacher contents were removed. In Runs 80 and 81 the line connecting the centrifuge to the leacher plugged with insols. It was necessary to disconnect the line and rinse it with water. This line plugging could be a very severe problem in the hot cell and will be investigated further.

The purgerator dip tubes (Ref. 35) for measuring the amount and specific gravity of the leacher contents developed restrictions or plugs in Runs 75, 78-81, and 84. No problems were encountered in runs that lasted less than 2-1/2 hr. The problem is thought to be the formation of a thorium nitrate precipitate around the end of the tube at the interface between the liquid and the dry nitrogen purge gas. The restrictions were easily removed by purging the dip legs with a small amount of water; in the longer runs, the restrictions necessitated removal several times during a run.

The solution thermocouple stopped working after Run 78. The thermocouple sheath had a welded end cap that was corroded away by the boiling Thorex. Therefore, there were no thermocouple readings for Runs

79-81 and 84. However, this posed no problem. The erratic movement of the ΔP pins plus the visual indication of reflux back to the leacher from the overhead condenser were utilized to note the time at which the solution began to boil. A thermocouple enclosed in a 304L sheath and welded with 308ELC wire will be utilized in future runs.

In Runs 79-81 and 84 several changes were made from the previous runs. Specifically, a different batch of burner ash was utilized (Fig. 48), and a series of corrosion coupons were installed in the leacher at three locations (see Ref. 4 for details). It is not clear to what extent these changes influenced the results depicted in Fig. 47 and Table 44.

The mother liquor analysis in Run 73 is suspected of being too high, thus the 113% material balance closure.

The thorium recoveries for Runs 81 and 84 in Fig. 47 appear to be low as a result of air sparge rate. This would, therefore, indicate that sparge rate is an important variable in the dissolution of this type of burner ash. This was not the case for the type fertile particle ash utilized in Runs 72-78 that were leached without the presence of whole fissile particles; i.e., the dissolution rate for only the crushed fertile particle ash was not a function of sparge rate (Ref. 34).

The problems with particle breakage discussed above necessitated the substitution of glass beads for the TRISO fissile particles. The main objective of this work was to investigate the effects of whole fissile particles on dissolution rates. As can be seen from Fig. 47, the use of glass beads to simulate the effect on dissolution of whole fissile particles seems acceptable. Further testing is under way and will be reported in the next Quarterly Progress Report.

5.2.4. Conclusions and Recommendations

1. Dissolution rates are reduced substantially when whole fissile particles are present in the crushed burned-back TRISO ThC_2 burner ash.
2. Further testing is required to substantiate and further analyze parameters affecting the dissolution of the combination whole fissile - crushed fertile burner ash.
3. Fissile material carryover is likely to be a problem in this flowsheet due to particle damage in the solid-liquid separation step. Further investigation is warranted to more closely define possible causes and the extent of carryover.
4. High solids concentrations in the leaching step are bound to lead to increased transport problems in downstream operations. Methods of minimizing these problems will be investigated.
5. Differential pressure legs, used to monitor specific gravity and weight of material, have some minor problems with plugging. Methods by which this can be overcome will be tested.

5.3. INVESTIGATION INTO THE POTENTIAL FOR U-232 CROSSOVER INTO FISSILE PARTICLES UNDER LEACH CONDITIONS

5.3.1. Summary

Refabrication potential of recycle U-235 without shielding is dependent on the extent of contamination of the U-235 fissile particle by the U-232 contained in the U-233 stream. It has been established that a U-233 contamination level not to exceed 100 ppm of U-232 would be required to achieve this end (Ref. 36). When BISO (or crushed burned-back TRISO) fertile particles are dissolved in the presence of whole TRISO fissile particles, a

potential exists for crossover of U-233 (and thus U-232) into the U-235 particles. The results of this investigation suggest that contamination resulting from crossover of U-233 into the U-235 particles during leaching can be maintained within the 100-ppm limit only under the "best of circumstances." The "best of circumstances" implies that lengthy solid-liquid contact times are avoided, uranium entrainment is low after washing of the particles, and particle defects/breakages are small or nonexistent. Further, the current work was performed with unirradiated particles, and the probability exists that crossover would be even greater for irradiated particles. Realistically, it appears from the data that uranium contamination by crossover can be expected to exceed the 100-ppm limit. It must be cautioned, however, that this test work was preliminary in nature, and additional testing should be conducted to provide more definitive results.

5.3.2. Experimental Procedure

To simulate the contamination of U-235 fissile particles by crossover of U-233, burned-back TRISO ThC_2 particles (used to represent the fissile particles) were leached in uranyl nitrate solutions (used to represent the U-233 particles) as a function of time and uranium concentration. After the appropriate leaching times, the particles were analyzed for uranium and the solutions for thorium content. The experimental flowsheet that was followed and the preparatory steps in the uranium analysis are shown in Fig. 49.

Two uranyl nitrate solutions were prepared with concentrations of 0.0425M and 0.1276M uranium (10 and 30 g/liter uranium, respectively). In comparison, FSV fuel data ranges from 1.5 to 9% uranium in the process stream (Ref. 37). This corresponds to uranium fuel concentrations of 3.6 to 23.6 g/liter uranium, respectively.

Fig. 49. Flow sheet for crossover studies

The TRISO coated particles were burned-back at 900°C for 24 hr in a muffle oven to remove the outer PyC coat. A 15% weight loss occurred. After burn-back, relatively clean unbroken particles were obtained by allowing separation to occur using an inclined plane.

Sample weights of 26.4 g of particles were refluxed for appropriate lengths of time in 100-ml uranium-Thorex solutions. The Thorex solution was 13M HNO_3 /0.05M HF /0.1M $\text{Al}(\text{NO}_3)_3$. After reflux for the times indicated in Table 45, the solutions were filtered and washed with 300 ml of H_2O . The last 30 ml of the wash was analyzed for uranium. The uranium concentration of the wash was never greater than 3 to 5 ppm. The particles were analyzed for uranium and the solutions for thorium. No attempt was made to analyze the SiC hulls for uranium that may have been tied up and not leached, since this amount was considered to be small.

The low-level uranium values reported here were determined by potentiometric titration (uranium reduced to U^{+4} and titrated to U^{+6} with $\text{K}_2\text{Cr}_2\text{O}_7$ solution). Alternatively, after some initial problems, an Arsenazo colorimetry method was used as verification for the titration method. Satisfactory correlation was obtained by the Analytical Chemistry Branch.

5.3.3. Results and Conclusions

The following inferences can be drawn from the data given in Table 45:

1. Uranium concentration within the particles after leaching is a function of leaching time (the longer the leach, the greater the uranium concentration). The disparity in the thorium concentration values after leaching does not permit any conclusions to be drawn concerning crossover as a function of uranium concentration of the solution.

TABLE 45
URANIUM AND THORIUM CONTENTS FOLLOWING LEACHING^(a)

Sample No.	Reflux Time (hr)	U Concentration of Particles after Leach (ppm)	Th Concentration of Solution after Leach	Initial U Solution Concentration (g/l)
1	20.0	4,5	56.96 g/l	10
2	2.5	37,45	35.0 ppm	10
3	7.0	34,58	6.0 ppm	10
4	24.0	84,83	17.0 ppm	10
5	7.5	106,107	0.91 g/l	30
6	17.0	123,139	1.40 g/l	30

(a) Experiment data:

1. Whole burned-back virgin TRISO particles; uranium content 10 ppm.
2. Uranium solution with no contact with TRISO particles; thorium content 4.5 to 5.9 ppm.
3. Leached whole burned-back TRISO particle solution analysis; 0.2 g/l Th/10-min leach and 0.32 g/l Th/30-min leach.
4. Last 30 ml of a 300 ml H₂O wash solution; uranium analysis 3 to 5 ppm.

2. The low value for uranium and the high values for thorium in sample 1 are the result of partial disintegration of the sample during leaching (magnetic stirrer was set too high, causing particles to be crushed). Subsequent washing of the sample rinsed away the uranium present.
3. For samples 2, 3, and 4, the thorium content of the particles is low, while the uranium content of the particles is high. The low thorium value suggests that the integrity of the inner coating is quite good, preventing thorium from being leached into the solution. The high uranium value suggests that the integrity of the SiC coat is questionable. Uranium apparently is being absorbed through or into cracks and/or fissures in the SiC coat.
4. For samples 6 and 7, the thorium and uranium values are both high. The integrity of the entire particle, both inner and outer coats, appears to be defective. The high thorium value suggests that thorium is being leached from the interior of the particle. The possibility does exist that this amount of thorium may be the result of particle breakage after burn-back. This, however, seems unlikely since a 10- and 30-min leach of particles in Thorex resulted in a thorium solution concentration of only 0.2 and 0.32 g/liter, respectively. Again, the high uranium value suggests that uranium is being absorbed into cracks or fissures in the particles and perhaps to a much greater depth than in the previous case.
5. To obtain an idea of the background contamination, uranyl nitrate solutions and ThC_2 particles having no prior contact with one another were analyzed for thorium and uranium, respectively. The thorium content of the uranium solution was 4.5 to 5.9 ppm and the uranium content of the ThC_2 particles was 10 ppm. It is felt

that these values are sufficiently low to not affect any of the results or conclusions given above. The data in Table 45 are the total metal content as analyzed with no correction applied.

In conclusion, crossover of uranium occurs by absorption through cracks or fissures in the particles and appears to be a function of the integrity of the particle coating. The uranium crossover is also a definite function of leach time and can be as great as 139 ppm for a 17-hr leach. The data are inconclusive with regard to crossover of uranium as a function of solution concentration. Further investigation would be required to clarify this point, as well as to further substantiate any of the data contained herein.

6. SOLVENT EXTRACTION

During the quarter, two run sets were made through the single-column solvent extraction system, and five run sets were made through the recently installed two-column system.

Data from two solvent washing and two coextraction, partitioning, partition scrubbing, uranium stripping runs are included here. Data from other runs already made will be forthcoming when analytical data are complete.

6.1. FLOODING TESTS - SOLVENT WASHING

The objective of these runs was to establish the capacity of the solvent wash columns with 30% TBP for both scrap recovery and HTGR reprocessing. No efficiency data were taken, since neither dibutyl phosphate nor analytical procedures are yet available.

A comparison between 10% and 23% free area nozzle plate cartridges is shown in Fig. 50. The planned operation at 50°C was not obtainable because of heat losses in the hot water exchanger system. (The lines have since been insulated, and future tests will be possible at column temperatures of 50°C.) The 23% free area cartridge will have more than adequate capacity for solvent washing in the cold pilot plant. Cartridge selection for solvent washing in fresh fuel scrap recovery and HTGR reprocessing will be made on the basis of these and future tests.

Fig. 50. Solvent wash column flooding curves for 30% TBP

Most of the solvent wash flooding tests were run using 0.27M Na_2CO_3 at an aqueous-to-organic flow ratio of 0.1. Because of concern that observed higher-than-expected flooding frequencies were the result of slow flooding brought on by this low flow ratio, tests were run at various ratios. The results of these tests are shown in Table 46. At a total volume velocity (sum of phases) of 1200 gal/hr/ft², phase ratios between 0.1 and 0.375 made little difference on the flooding frequency. However, at higher volume velocities, the aqueous flow was limiting; the column could not be stabilized at aqueous phase volume velocities of 450 when the sum of phases approached 1900.

6.2. URANIUM-THORIUM COEXTRACTION, THORIUM PARTITIONING, PARTITION SCRUBBING, AND URANIUM STRIPPING

The objective of these runs was to provide pulse column efficiency and capacity data for uranium-thorium coextraction, thorium partitioning, partition scrubbing, and uranium stripping. These data are applicable to solvent extraction in fresh fuel scrap recovery, the Idaho HTGR demonstration plant, and commercial HTGR reprocessing.

The flowsheet tested is as shown in Fig. 5-23 of Ref. 1, with the exception that the equipment to add the side streams to the extraction column was not yet installed.

Table 47 shows the stream analyses for each type operation and the flow ratio of each stream.

Table 48 shows the flooding data and the height equivalent to a theoretical stage (HETS) when operating at 80% of that flooding frequency.

Run 7 was intended to be run at 175% of the flow rates of Run 6, but in some cases the columns could not be operated at the higher flows and Run 6 conditions were duplicated.

TABLE 46
FLOODING DATA AS A FUNCTION OF PHASE RATIO^(a)

Volume Velocity (gal/hr/ft ²)	Aqueous-to- Organic Flow Ratio	Flooding, Amplitude x Frequency	Temp (°C)
1200	0.1	112	41
1200	0.22	114	39
1200	0.375	109	38
1600	0.1	98	38
1600	0.375	87	37
1745	0.2	93	38
1890	0.3	<60	38
2000	0.1	>100	39

^(a) 23% free area, 1/8-in.-diameter hole nozzle plates, 2-in. plate spacing, organic, continuous.

TABLE 47
ANALYTICAL DATA AND STREAM FLOWS - RUNS 6 AND 7

Stream	Stream No. in Fig. 2, Ref. 38	Run 6 Analyses			Run 6 Flow (ml/min)	Run 7 Analyses			Run 7 Flow (ml/min)
		Th(g/l)	U(g/l)	HNO ₃ (M)		Th(g/l)	U(g/l)	HNO ₃ (M)	
1AF	1	339	34.7	0.85	110	358	35.5	0.98	180
1AS	2	-	-	0.98	115	-	-	1.01	210
1AX	3	33% TBP	-	0.26	1030	34% TBP	-	0.25	1725
1AP	5	32	3.2	0.21	-	34	3.0	0.21	-
1AW	4	2.1	0.0002	1.1	225	2	0.007	1.1	390
1AP (avg)	5	32 ^(a)	2.94	0.15	1000	32	2.9	0.22	990
1BX	6	-	-	0.18	620	-	-	0.19	590
1BXT	7	53	0.58	0.47	-	52.5	0.49	0.54	-
1BU	8	0.03	3.0	0.013	-	0.004	2.7	0.017	-
1BXT (avg)	7	44.4	0.49	0.5	615	45	0.72	0.54	950
1BS	9	33% TBP	-	-	205	35% TBP	-	-	325
1BT	10	36.0	0.004	0.5	-	36	0.009	0.50	-
1BSU	11	23	1.3	0.1	-	27	2.2	0.2	-
1BU (avg)	8	-	2.0	0.07	1020	-	-	-	1650
1CX	12	-	-	0.009	490	-	-	-	900
1CW	14	-	0.002	0.004	-	-	-	-	-
1CU	13	-	4.0 ^(a)	-	-	-	-	-	-

^(a) Calculated by stream flow and material balance.

TABLE 48
COLUMN HETS AND FLOODING DATA - RUNS 6 AND 7^(a)

Column	Purpose	Run 6				Run 7			
		Volume Velocity (gal/hr/ft ²)	Flooding Frequency (cycles/min)	Continuous Phase	HETS (80% Flooding) (ft)	Volume Velocity (gal/hr/ft ²)	Flooding Frequency (cycles/min)	Continuous Phase	HETS (80% Flooding) (ft)
1A	Extraction	910	126	Organic	2.7 Th	1550	120	Organic	2.6 Th
1BX	Partition	1190	120	Organic	3.5 Th (5) ^(b)	1190	120	Organic	3.0 Th
1BS	Partition scrub	595	80	Aqueous	2.6 U (3.3) ^(b)	925	64	Aqueous	2.6 U
1C	Strip	1050	35	Aqueous	4.7 U	1900	>88	Organic	-

^(a) 23% free area, 1/8-in.-diameter hole nozzle plates, nozzles down, 2-in. plate spacing (18-ft length), 1-in. amplitude.

^(b) Numbers in parentheses are HETS values for additional stages for columns longer than 18 ft.

The HETS were calculated from McCabe-Thiele diagrams using the best estimate available for equilibrium lines. The HETS data are average numbers for the length of column used; additional column length will change the average HETS. Some column profile analyses were taken, and the estimated HETS for added column length are also shown in Table 48 where available.

The salting acid for the bottom of the extraction section was added with the solvent. It is not known what effect this has on flooding or HETS, but it will not be highly significant.

7. SYSTEMS DESIGN

7.1. PROTOTYPE SIZE REDUCTION SYSTEM

The prototype size reduction system was initially designed for Cell No. 2 at ACC. Certain salient remote maintenance features were included in the design, but the scope was limited. The UNIFRAME system design was completed, procurement of major mechanical components was under way, and the structural framework design was being reviewed.

This direction was officially changed August 19, 1974 after a joint meeting with GA, ACC, and the AEC. General Atomic was directed by RDD to design for remote maintenance, and the restriction of installing the crusher in Cell No. 2 was no longer a design requirement.

Based on these new guide lines, GA has been redesigning the system to demonstrate remote maintenance. This effort has uncovered areas of interference within the welded UNIFRAME structure. Modifications to the double-roll crusher and drive, the drives for the two jaw crushers, and the vibrating-screen separator are required. Work continues toward resolution of these problem areas to determine whether a design analysis for a new UNIFRAME configuration is warranted.

Automatic lubrication systems are being investigated, and outside vendors have been contacted. Initial results indicate a remote automatic lubrication system is feasible.

7.2. PROTOTYPE PRIMARY BURNER

Conceptual and final designs of the prototype primary burner have been completed. Detail design drawings are in progress. Nine drawings illustrating the vessel assembly were completed and transmitted to ACC and the Ralph M. Parsons Company for review. Drawings of the burner assembly have been submitted to the GA Stress Analysis Group.

Meetings were held between GA and the Gray Tool Company (Houston, Texas) concerning design of the high-temperature remotely operated flange. The Gray Tool Company will design and fabricate the flange to GA specified requirements (ASME Section VIII, Division 1).

Meetings have been held with insulation suppliers regarding insulation required around the burner tube. The Glas-Rock Company of Atlanta, Georgia was contacted and can furnish an insulating material suitable for GA requirements. This material is now being designed into our system.

Different heating/cooling concepts have been studied relative to the induction coil, and suppliers have been contacted. The Tocco Company of Los Angeles, California was selected to fabricate the coil assembly. Tocco has evaluated the GA design and can meet the design requirements.

7.3. PROTOTYPE SECONDARY BURNER

Conceptual and final designs of the secondary burner have been completed, and detail design drawings are in progress. Four drawings illustrating the vessel assembly were completed and transmitted to ACC and the Ralph H. Parson Company for review.

Based on information obtained from meetings held with the Gray Tool Company on the primary burner, semi-remote flanges are being incorporated in the design of the secondary burner assembly.

Conceptual design of the induction heating coil assembly has been completed and final design initiated. The Tocco Company of Los Angeles, California has been selected to fabricate the coil.

7.4. PROTOTYPE SOLIDS HANDLING

The filter assembly conceptual and final designs for the prototype primary burner and the prototype secondary burner were completed. The conceptual and final designs of the feed hopper for the primary burner were also completed and the pneumatic product feeder design for the prototype secondary burner is complete.

7.5. PROTOTYPE PLANT SYSTEMS OPERATIONS

A conceptual arrangement of the crusher and burner units was created to provide an overview of the system.

Plan and elevation views depicting the approximate sizes and relative locations of each piece of equipment are shown in Fig. 51, Sheets 1 and 2, respectively. An elevation plan of the simulated "hot-cell" wall for viewing of remote operations is shown in Fig. 51, Sheet 3.

Structural loading diagrams defining support structure and working platform requirements are in preparation for submittal to plant engineering. Work platforms will be extended to enclose the crusher area, providing full access capability.

7.6. REMOTE OPERATIONS AND MAINTENANCE OF PROTOTYPE PILOT LINE

To demonstrate prototype component configuration and adaptability for remote installation, operation, maintenance, removal, and/or disassembly, the pilot plant will simulate hot cell operating conditions. An existing 20-ton electric overhead traveling (EOT) crane and fork-lift tractors are available to simulate "heavy-weight" remote tools.

Fig. 51. Prototype plant arrangement, burning and crushing
(Sheet 1 of 3)

Fig. 51. Prototype plant arrangement, burning and crushing (Sheet 2 of 3)

ELEVATION
 SECTION B-B REF SHT. 1

Fig. 51. Prototype plant arrangement, burning and crushing
 (Sheet 3 of 3)

The components will be arranged in plan (Fig. 51, Sheet 1) to require a minimum space envelope and in elevation (Fig. 51, Sheet 2) to facilitate maximum use of gravity for solids transfer.

A wall of light structural construction may be used to partially enclose the crushing and burning operations to simulate a hot cell (Fig. 51, Sheet 2). The wall would have two or more simulated hot cell windows with operator platforms, located approximately opposite the location of the primary prototype burner.

Personnel access into the equipment area would be by man-doors in the enclosure wall. A double-leaf hinged door opening the full height of the wall (no lintel across the door frame) would facilitate installation and removal of large equipment by the EOT crane and the fork-lift tractor.

As a design criterion, a semi-remote philosophy will be applied in general, with some specific areas developed for fully remote operation. The latter areas include a fully remote separation of the flanged connection of the primary burner vessel at the distributor plate (see Fig. 21 in Section 4), remotely operated latching/unlatching of components of the suspension system of the induction heating coil, opening and closing of the clamshell-type insulating shrouds on the burner, elevating and pivoting supports for filters, hoppers and cyclones, plus the usual remote application of valves, positioning stops, fail-safe keepers, etc.

Equipment will be arranged and supported so as to be readily accessible by the existing EOT crane and by personnel. Where possible, automatic guiding and positioning will be included and securing will be effected with a minimum of bolts and other permanent-type fasteners. Wide use will be made of male-female type slip-fit securing devices for maximum ease of installation and removal or disassembly. Component equipment

design will incorporate a "unit" concept, permitting installation and removal as subassemblies or complete assemblies as warranted. This will allow substitution when time is of the essence or repair is too difficult.

Special remote handling aids and tools, such as dollies with elevating platforms on tracks, lifting spreaders, aligning fixtures, slide tracks, jacks, and impact wrenches, will be developed or adapted by modifying purchased items.

In summary, simplicity of detail, reliability of operation, applicability of materials, economy of manufacture, ease of installation, and accessibility for maintenance and repair, as well as functional adequacy, are the main considerations being applied in designing for remote operation.

7.7. E-BUILDING

The floor plan of the E-Building was revised to show proposed locations for the prototype pilot systems, a new control room, office space, a new wash and locker facility, a simulated hot-cell enclosure, extension of solvent extraction operations, consolidation of the shop areas, and a storage enclosure outside the south end of the high-bay area (Fig. 52).

Fig. 52. E-building layout

8. ALTERNATE HEAD-END TECHNIQUES

8.1. INTRODUCTION

The reference flowsheet for reprocessing of HTGR fuels includes fluidized-bed burning of crushed fuel elements to remove moderator graphite and pyrolytic carbon prior to dissolution of the fuel materials and fission products. During this combustion, quantities of CO_2 will be generated and released. A small amount of carbon-14 will be present in the carbon dioxide, which will contribute to the radioactive material release from a reprocessing plant. Methods that will reduce the quantity of this material released are being investigated.

Carbon-14 is generated in an operating HTGR by neutron interactions with carbon-13, which constitutes 1.1% of natural carbon, and with the impurity nitrogen-14 contained within core materials. Elimination of nitrogen from the manufactured fuel or segregation of constituents of the irradiated fuel containing most of the carbon-14 are two possible methods of reducing the amount of carbon-14 released.

In order to direct the investigation of methods to reduce impurity levels of nitrogen in the fuel, it is necessary to determine the levels now found in manufactured fuel for HTGRs. A subcontract was let to Dow Interpretive Analytical Services of Dow Chemical U.S.A. of Midland, Michigan to develop methods for analysis of nitrogen in fuel rods and in graphite.

8.2. SUMMARY OF RESULTS

Initial experiments were performed to analyze for "physically" and "chemically" adsorbed nitrogen in fuel rods and graphite. "Physically"

adsorbed nitrogen was defined as that which would be released on crushing to a fine powder and "chemically" adsorbed nitrogen as that which would be released upon heating the crushed material.

An apparatus was constructed in which samples were crushed in a helium-filled container connected to a gas chromatograph. Any gas released was purged from the container through the gas chromatograph. The sample was then heated to 425°C and the off-gas was passed through the chromatograph for analysis. Results were inconclusive; no significant amounts of nitrogen were detected either immediately upon crushing or after heating to 425°C.

Additional experiments were conducted in which known quantities of nitrogen were introduced into the system and were adsorbed by the crushed graphite, after which the sample was ground and reheated. Even with a known quantity of nitrogen added as a spike, no significant amount of nitrogen was detected after heating. This indicates that nitrogen which is adsorbed by graphite is not removed by heating to 400°C. Experiments at higher temperatures were not possible due to thermal limitations of the experimental apparatus.

It was concluded that with the experimental processes used, no distinction could be made between "physically" and "chemically" adsorbed nitrogen. Methods to determine the total nitrogen in graphitic materials are inert gas fusion and complete combustion followed by gas chromatography.

Results from samples sent to LECO for analysis by the inert gas fusion method are summarized in Table 49. Samples of fuel rods containing ThO_2 particles and sample graphite crucibles were prepared at General Atomic for this analysis. The fuel particles in the fuel rods were produced using either nitrogen or argon as the levitating gases in the coating process and were exposed only to nitrogen or argon during processing and fuel rod assembly.

TABLE 49
TOTAL NITROGEN ANALYSES BY LECO

Sample No.	Description	Processing	Nitrogen Content
RA 1019-1-A	Fuel rod	All argon	449
-B			173
-C			92
-D			<u>299</u>
			253 avg
RA 1019-1C-A	Graphite crucible	All argon	200
-B			206
-C			480
-D			<u>397</u>
			321 avg
RA-1022-1-A	Fuel rod	All nitrogen	78
-B			205
-C			266
-D			<u>163</u>
			178 avg
RA 1022-1C-A	Graphite crucible	All nitrogen	276
-B			693
-C			700
-D			<u>252</u>
			480 avg
Blank			5

No correlation is apparent in Table 49 between samples produced under each atmosphere and the data spread is extremely broad. The general levels measured in these samples do not represent those that might be found in an operating HTGR since the samples had not been pretreated to simulate heatup to operating conditions in the reactor. Samples have been prepared and subjected to a simulated heatup but have not been analyzed to date. These pretreated samples are currently being stored at General Atomic in sealed metal cans filled with argon. A future decision will be made as to whether these samples should be analyzed by inert gas fusion.

The development of a method to perform gas chromatography on combustion products was not successful. A molecular sieve trap was constructed to absorb the nitrogen released. However, CO₂ had to be removed ahead of the trap to prevent its freezing in the trap at liquid nitrogen temperature. Methods that proved successful in removing the CO₂ also absorbed most of the nitrogen. No successful technique for separating these two components was found.

8.3. CONCLUSIONS AND RECOMMENDATIONS

Analytical results indicate that nitrogen adsorbed in graphite is not desorbed at temperatures up to 400°C. Releases, if any, at HTGR core temperatures could not be determined due to limitations of the apparatus.

Data on total nitrogen content in fuel and graphite samples by the inert gas fusion method indicated a wide spread in composition. Fuel samples that had not been exposed to a temperature cycle simulating reactor startup showed no correlation between fuel prepared in an argon atmosphere and that prepared in a nitrogen atmosphere.

The mechanisms of nitrogen interactions with fuel material after manufacture and during heatup in the reactor are extremely difficult to determine. Future analytical work will focus on measuring actual carbon-14 concentrations in irradiated fuel samples. Attempts will be made to correlate carbon-14 concentrations in these samples to various process parameters.

9. CONCEPTUAL DESIGN OF AN HTGR FUEL COMMERCIAL RECYCLE PLANT

Work was initiated with the Ralph M. Parsons Company of Pasadena, California, on a conceptual design of a commercial plant for the recycle (reprocessing and refabrication) of HTGR fuel. This effort is jointly funded by the Thorium Utilization Program and General Atomic Company.

The conceptual design is based on the following basic plant requirements:

1. The plant will be capable of reprocessing 50,000 spent HTGR fuel elements per year based on 80% availability and 90% efficiency factors.
2. The plant will be capable of refabricating 21,500 recycle U-233 and 3,800 recycle U-235 fuel elements per year based on 80% availability and 90% efficiency factors. Separate remotely operated refabrication lines will be used for the two types of recycle fuel.
3. The fuel contains BISO coated fertile particles and TRISO coated fissile particles. U-235 fissile particles in initial and makeup elements will be processed to recover the uranium for recycle. U-235 fissile particles in recycle fuel elements will be retired. U-233 fissile particles will be processed by combining the dissolved recycle U-233 with the bred U-233.
4. The plant will have the capability of storing 15,600 spent fuel elements and 5,900 refabricated fuel elements (5,000 recycle U-233 elements and 900 recycle U-235 elements).

5. Fluidized bed burners will be used for graphite burning.
6. The acid-Thorex process will be used for U-233 recovery, and a modified Purex process will be used for recycle U-235 recovery.
7. Off-gases will be treated for krypton, radon, and tritium removal.
8. Capability will be provided for the storage of 1 year's accumulation of high-level liquid waste (HLW). The HLW will then be calcined, packaged in high-integrity containers, placed in interim storage, and finally shipped to a federal repository. Retired U-235 fissile particles and SiC hulls will be similarly packaged, stored, and shipped.
9. Fissile particle preparation will utilize the resin process. TRISO coatings will be used on the fissile particles. The hot injection process with pitch binder will be used for fuel rod fabrication.
10. Coated fertile particles, graphite fuel blocks, and burnable poison rods will be obtained from the fresh fuel plant.

To guide the conceptual design effort, the following manuals and criteria documents were prepared:

1. Design Criteria: Commercial Reprocessing Plant: Requirements, DC-202001.
2. Design Criteria: Commercial Refabrication Plant: Requirements, DC-203001.
3. Design Criteria: Commercial Recycle Plant: Safety Requirements, DC-208001.

4. Design Criteria: General Design Philosophy, DC-209501.

As a means of implementing the design, the following 20 process and support systems were established and defined:

1. Spent fuel element handling
2. Refabricated fuel element handling
3. Head end
4. Solvent extraction
5. Fissile particle fabrication
6. Fuel element fabrication
7. Scrap recovery
8. Ventilation and cooling
9. Off-gas
10. Liquid waste
11. Solid waste
12. Decontamination and maintenance
13. Process support (analytical)
14. Process support (cold chemical and plant control)
15. Utility systems

16. Plant safety (security and protection)
17. Safeguards
18. Site
19. Process buildings
20. Non-process buildings

For most of the above individual systems, block flow diagrams, system descriptions, and design criteria have been prepared or are in the final stages of preparation. These serve as the basis for the preparation of process flow diagrams and piping and instrument diagrams. Preparation of the process flow diagrams and piping and instrument diagrams is well advanced, with most of these diagrams in some stage of drafting or review. Material balances are included with the process flow diagrams.

Plant arrangement work has been initiated. In the areas of spent fuel receiving, spent and refabricated fuel storage, and refabricated fuel shipping, conceptual arrangement drawings are in advanced stages of preparation. The layout of the fuel storage area is based on the fuel shipping containers, which also serve as the fuel storage containers. Very preliminary drawings have been prepared depicting initial concepts on plant and site arrangement.

The next stage of the conceptual design is the development of the equipment specifications. The initial effort in this stage is the preparation of equipment lists. Such lists have been prepared for the head-end and solvent extraction systems.

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